Appendix B

Field Sampling SOPs

Field Notes/Records

FIELD NOTES/RECORDS

Field notes must be copied to the Project Manager, or the Project Engineer/Geologist when the project schedule dictates. Don't invest time in editing or rewriting them; the notes should have been taken throughout the day and, therefore, they should be a sufficiently accurate record in their original form.

By providing the field notes in a timely manner, the Project Manager will have the field information and can begin to get any of his questions about the notes answered right away.

NOTES MUST INCLUDE:

- 1. Project Name
- 2. Project Number
- 3. Originator's Name
- 4. Date
- 5. Location of Field Activity
- 6. Materials and Equipment
- 7. Weather limited information (sunny, overcast, humid, precipitation, wind, approximate temperature)
- 8. Methods, Brief "according to work plan". If deviation from plan, you must note what was done differently, why, and the results. You must note whether the deviation was called for by you, or a departure by the contractor. If by the contractor, whether you have approved or disapproved it.
- 9. Problems Encountered how they were dealt with. Problems include equipment malfunction, delays, unsafe working conditions or procedures, departure from the Health & Safety Plan, attitudes/comments of workers/visitors, weather adversely affecting the work, and inspected work found to be unsatisfactory. Include a sketch of an unusual procedure, if helpful.

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- 10. Site Visitors who and when, their comments.
- 11. Location Descriptions wells, borings, and sampling stations. Distance tie-in to two fixed site facilities.
- 12. Summary of Work Accomplished.

Notes must reference any forms used for documenting calculations, location descriptions, depth measurements, and time and materials.

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Elevation Surveys for Monitor Wells

ELEVATION SURVEYS FOR MONITOR WELLS

EQUIPMENT LIST

standard surveyors level

stadia rod

· two persons

hand-held tape in 0.01-foot increments

· well lock keys

· tools to open well caps

· survey forms

tripod

· calculator

JOB DESCRIPTION

Obtain survey elevations for wells.

EXPECTATIONS

- Shoot and record well elevations at the top of casing.
- Shoot and record ground elevation at each well location.
- Tie all loops together and to reference datum.

The data generated from the survey is used to obtain accurate elevations which are necessary to gain a common reference point from which groundwater elevations, well screen elevations, cross-section maps, elevation contour maps, and more can be generated and compared to each other.

Before going into the field, decide with the person who has requested the work what the reference point should be. If the reference point is to be taken from a U.S.G.S. datum, it is necessary to know exactly where that benchmark is before beginning the survey. If a relative datum (e.g., 100.00 feet) is used, choose a benchmark that is a part of a permanent structure. Accurately detail the location chosen so that results may be duplicated in the future if necessary. An example of a good permanent benchmark may be a specific corner of a concrete foundation of a specific building, or a specific point on a fire hydrant.

The basic procedure is (refer to Example 1):

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- 1. Begin the survey by recording the elevation and location of the reference point.
- 2. Set up the tripod and surveyor's level at the first station and level the instrument. Check instrument level by rotating 180°; relevel if necessary.
- 3. Have one party hold the stadia rod on the reference point after the level and tri-pod have been set up.
- 4. Record the elevation shot in the +S column. By adding the +S to the known elevation, the height of the instrument (HI) will be determined:

Elevation
$$+ S = HI$$

- 5. The rod man moves in the general direction of the well location and chooses a good turning point. The turning point must be well defined and solid, such as a rock, tree, root, or the top of a screw driver driven solidly into the ground.
- 6. Record the reading at the turning point as a -S on the form. The -S reading is subtracted from the HI to give the elevation of the turning point:

- 7. The instrument man now moves the tri-pod and level in the general direction of the well.
- 8. The new reading is recorded as a +S.

Elevation at
$$TP + S = New HI$$

- 9. The procedure is repeated until a -S reading can be taken at each well top-of-casing and at each ground level at the well.
- 10. It may be necessary to use several turning points and loops to obtain all the data required. Be sure they are all tied together.
- 11. Once all the elevations have been recorded, close the loop. The rod man and instrument man proceed as before back to the original reference point. The loop is closed when the rod man returns to the original point of reference and that elevation is again recorded.

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12. Clean the well cap threads with a wire brush and lubricate with teflon paste or beeswax before

replacing.

13. Refer to the elevation survey illustration for a simple example.

ACCURACY

Each elevation measurement must be recorded to the nearest 0.01 foot. An elevation survey is of

acceptable accuracy if the beginning and final elevation are within ± 0.03 feet.

Check the work when the survey is complete. Make sure the locations of the benchmarks are

described in detail. Describe any difficulties which may have had an effect on the data. Turn in

the paperwork when it is completed and legible to the person who requested it. Keep records for

personal files.

METHOD REQUIREMENTS

• If the rod cannot be positioned at the TOC, measure and record the elevation difference from

TOC to the point where the rod was positioned.

• Rock the stadia rod toward and away from the instrument man at each location. The

instrument man should record the lowest elevation reading as the rod is rocking. The lowest

reading indicates when the rod is straight up and down.

• Use the top of casings (TOC's) as turning points. If this can be done, it will be possible to

mathematically verify the results from the office.

IMPORTANT TERMS

BENCHMARK (BM):

A definite point of known or assumed elevation not subject to change. Permanent benchmarks

have been established throughout the United States by the U.S. Coast and Geodetic Survey and

the U.S. Geologic Survey (U.S.G.S.). Benchmarks are used as starting points for surveys

requiring a common reference point.

SURVEY FIELD NOTES:

The record of the work performed. Typical entries should be:

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date

description of weather

names of individuals in the party and their responsibilities

the instrument and methods being used

LEVEL NOTES:

The standard form for recording stations and elevations taken (see example).

PLUS SIGHT (+S):

A rod reading taken on a point of known or assumed elevation. The plus sight added to the elevation at the point gives the height of the instrument.

MINUS SIGHT (-S):

A rod reading taken on a point where elevation is to be determined. HI - S = the elevation of the point.

HEIGHT OF INSTRUMENT (HI):

The elevation of the line of sight when the instrument is level.

TURNING POINT (TP):

A solid, well-defined point on which the rod is set while the instrument is being moved from one location to another. A minus sight is taken on a TP from the first level set-up to determine its elevation. A plus sight (+S) is taken on a TP from the second set-up to determine the new height of the instrument.

TOP OF CASING (TOC):

The top of casing or top of threads is the uppermost point of the well casing.

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Monitoring Well Sampling With A Bailer

MONITORING WELL PURGE AND SAMPLING WITH A BAILER

The objective in well sampling is to obtain a representative sample of the ground water from the formation where the well screen has been placed.

JOB DESCRIPTION:

Obtain ground water samples from the specified wells.

TASK-SPECIFIC EQUIPMENT AND MINIMUM INFORMATION NEEDED:

· detailed well location map

water level tape (electric or steel)

· order of the well sampling

well pumps if necessary

• previous water level data

polypropylene rope

· disposable gloves

bailers

· sample bottles

· container for purge water (if required)

· calculator

well keys

• $V = Hr^2 (0.163)^a$

total well depth data

^aRefer to guideline for calculating the volume of standing water in a well casing.

OR alternatively if the filter pack volume is to be considered: r = auger O.D./2 in inches.

EXPECTATIONS:

All water levels will be taken prior to sampling.

All purge volume data will be recorded.

Standard decontamination procedures will be followed.

Noticeable discoloration or odor in the water will be reported.

Each sample requested will be collected.

PROCEDURES:

- 1. All the wells of a cluster to be sampled are uncapped. Care must be taken not to mix the caps up. The caps should be placed near the well on a clean area, such as a small piece of plastic. Inspect the condition of the well(s).
- 2. Take a round of water levels.
- 3. Consult the field sampling plan for purging reqirements. Calculate and record the volume of water in the casing, with or without the filter pack volume, depending on the filed sampling plan. Record the needed purge volume; typically, this is three times the volume of water in the casing and filter pack or casing alone.
- 4. Purge the well with a clean or dedicated bailer and a new length of polypropylene rope. Concentrate the purging effort at the air/water interface.
- 5. Record the amount of water actually purged and what was done with the purge water. Record the method of purging, and the type of bailer used (Teflon or stainless steel).
- 6. Collect the ground water sample with the bailer.
- 7. Fill the sample container accordingly.
- 8. Seal the container.
- 9. If the container is a VOC vial, fill the vial completely until a convex meniscus is formed at the top of the vial and cap quickly so that the vial contains no headspace. Turn the full container upside down and tap it lightly. Watch for air bubbles. If air is present in the bottle, discard and resample the well.
- 10. Label the sample bottle(s) and place in a cooler with ice for transport to the laboratory.
- 11. Follow standard decontamination procedures if bailer is to be re-used.
- 12. Dispose of the used rope.

In wells which do not readily recover, it may be impractical to purge three well casing volumes prior to sampling. In these cases, a field judgment must be made as to what is a

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"reasonable" amount of time to spend in securing the sample. In a well that can be bailed dry, it is acceptable to purge one casing volume, wait for the well to recover, and take a sample. Keep good records of the volume of water actually purged and estimate the recovery time for the well. The purpose of purging is to remove all the static water from the well. In a well which is bailed dry, that objective is obtained after one well casing volume is removed.

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Soils Classification

SOILS CLASSIFICATION

There are several different soil classification systems. In order to maintain a level of consistency and conformity with widespread practices, we have adopted the Unified Soils Classification System (USCS). Use of the generalized symbols of the USCS facilitates soil correlation's and the production of geologic cross sections.

Most soil descriptions are written in the field based on visual and manual observations, but if a more accurate description is required, than the laboratory analyses that are required to definitively classify the soil sample according the USCS are a sieve analysis and the Atterberg limits.

There are four major soil divisions in the USCS:

- 1. Coarse grained,
- 2. Fine grained,
- 3. Organic soils, and
- 4. Peat

Soils too large to pass through a 75 mm sieve (about 3 inches) are considered "oversized" material according to this system. These "oversized" materials are boulders and cobbles.

Each soil description should follow this general order:

- 1. Texture
 - a. basic
 - b. modifying
- 2. Consistency
- 3. Color
- 4. Moisture content

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After the moisture or water content of the soil has been described, further observations--such as odor, presence of roots or debris, or any other notable observations--are recorded.

Each of these items is described in more detail below.

1. Texture

a. Basic Texture

USCS <u>Division</u>	Basic Texture or Description to Use	Particle Diameter Size	Common Comparison
Oversized	Boulder	larger than 12 inches	
Soils	Cobble	3 to 12 inches	
Coarse grained	Coarse gravel	3/4 to 3 inches	
Soils	Fine gravel	4.75 mm to 3/4 inch	pea to large marble
	Coarse sand	2 to 4.75 mm	pepper to pea size
	Medium sand	.425 mm to 2 mm	pencil lead to pepper
	Fine sand	.075 to .425 mm	table sugar
Fine grained Soils	Silt	<.075 mm	powdered sugar
	Clay	<.075 mm	individual grains are not visible
Highly Organic	Peat	organic, fibrous or	
Soils		amorphous textured	
		vegetable tissue	
	Organics	defined as muck, coal, etc.	

Clay is distinguished from silt by plasticity. Silt is non plastic or very slightly plastic and exhibits little or no strength when air dry (plasticity index <4). Clay can be made to exhibit

plasticity (putty-like properties) within a range of water content, and it exhibits considerable strength when air dry (plasticity index >4).

To distinguish clay from silt in a moist field sample, clay will ribbon down to a thickness of approximately 1/32 of an inch and can be molded into any shape. As the silt or sand content in a clay increases, so does the ribbon thickness and molding the sample becomes more difficult. A moist silt sample will ribbon to only 1/4 inch or greater in thickness and is more difficult to mold.

In wet soils, clay, when rubbed into the palm of a hand, is very difficult to rub off. Clay will hold its water content when shaken. When a wet silt dries in the hand, it can be rubbed off readily and a wet silt sample will puddle readily when shaken. See Attachments 1 and 2 for field guidance to distinguish silts from clays.

b. Modifying Texture

Estimates of the modifying texture are given using the following adjectives:

For sands and finer grained particles:

DescriptiveWord	Estimated Percentage
Trace	less than 10%
Little	10 to 30%
Some	30 to 45%
And	45 to 50%

For particles coarser than gravels (i.e., cobbles and boulders), the adjective "occasional" may be used to describe their percentage. An estimate of the maximum grain size should also be stated in the description for coarse gravel and larger grain sizes.

2. Consistency

The consistency of sands or gravels is described adequately by the blow counts required to drive the split-spoon sampler. Therefore, if the blow counts are recorded, the description of consistency may be omitted. For fine grained soils (i.e. clays), the description of consistency

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in addition to the recorded blow counts is more informative. For clay, use the following descriptions to define the consistency.

<u>Description</u> <u>Criteria</u>

Very hard Thumbnail will not dent

Hard Thumb will not indent soil but readily indented with thumbnail

Firm Thumb will indent soil about 1/4 inch

Soft Thumb will penetrate soil about 1 inch

Very soft Thumb will penetrate soil more than 1 inch

The consistency of clays can be defined in the laboratory by the results of the unconfined compression test.

Plasticity

Refer to Table 4 and especially the section on *toughness* for a practical approach to estimation of plasticity (consistency near plastic limit) in the field; and based on that, to assign "H" or "L" to cohesive organic samples.

3. Color

Color is useful in distinguishing soils similar in geologic origin and post-origin processes. Record the color of the soil as you see it. The color may be modified by an adjective (e.g. light brown). If there are two major but distinct colors in the soil, describe the color as mottled or variegated (e.g., gray mottled brown).

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4. Moisture Content

Describe the water content of every soil type encountered. Three main adjectives to use are:

Water Content

Sample Characteristics

Dry

Powdery or hard

Moist

Plastic or containing some liquid

Wet

Saturated or puddles when shaken

Again, modifiers can be added to describe varying degrees of moisture content.

Once the soil boring is complete and all the soil types have been described, assign the appropriate USCS symbol to each soil description on the soil log so that similar soils can be clearly grouped together to represent the major soil layers present at the site (Tables 1, 2 and 3).

Examples:

Soil Description USCS Symbol

SAND, fine to medium, some silt, trace clay, light brown, wet

SM

CLAY, some silt, trace medium to fine sand, soft, brown mottled gray, ML/CL moist (trace rootlets)

SILT, trace fine sand, yellow-brown, dry

ML

SAND AND GRAVEL, brown, dry

SW/GW

CLAY, little silt, trace fine to medium sand, occasional cobble (6"), very CL hard, gray, slightly moist

PEAT, brown, very soft, wet

PT

SILT, some clay, little organics, very soft, moist (decaying odor)

OL/OH

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A borderline symbol is two symbols separated by a slash, for example OL/OH. A borderline symbol is used to indicate a soil that has been identified as having properties that do not distinctly place the soil into a specific group.

Each log should be accurate and detailed. The frequency of soil sampling should be reflected in the detail of the well/boring log. The log for a borehole sampled continuously will be much more detailed in both soil descriptions and depths than a log for the borehole in which no soil samples were collected.

Well/boring log forms must be completed for every well or boring installed, regardless of depth or method used, even if it has not been specifically requested.

References:

1991 Annual Book of ASTM Standards, Volume 04.08 D 2488-90 "Standard Practice for Description and Identification of Soils (Visual-Manual Procedure)

An Introduction to Geotechnical Engineering Robert D. Holtz, William D. Kovacs, Prentice-Hall Civil Engineering Mechanics Series

AGI Data Sheets, 3rd Edition Compiled by J.T. Dutro, Jr., R.V. Dietrich, R. M. Foose, American Geological Institute 1989, Data sheets 29.2 and 38.1

Earth Manual, A Water Resources Technical Publication, 2nd ed., U.S. Department of the Interior, United States Government Printing Office Washington 1974. Reprinted in 1980.

ATTACHMENT 1

Field Identification Procedures for Fine Grained Soils¹

These procedures may be performed on the minus No. 40 sieve size particles, approximately 1/64 inch for field classification purposes; screening is not intended, simply remove by hand the coarse particles that interfere with the tests.

DILATANCY: (Reaction to shaking)

Take a small amount of moist soil. Add enough water, if necessary, to make the soil soft but not sticky. Place the soil in the open palm of one hand and shake horizontally, striking vigorously against the other hand several times. A positive reaction consists of the appearance of water on the surface of the soil which changes to a livery consistency and becomes glossy. When the sample is squeezed between the fingers, the water and gloss disappear from the surface, the sample stiffens, and finally it cracks or crumbles.

The rapidity of the appearance of water during shaking and of its disappearance during squeezing assist in identifying the character of the fines in a soil.

Very fine clean sands give the quickest and the most distinct reaction whereas a plastic clay has no reaction. Inorganic silts show a moderately quick reaction.

DRY STRENGTH (Crushing characteristics)

Mold a small sample of soil to the consistency of putty, adding water if necessary. Allow the soil to dry completely by sun or air drying, and then test its strength by breaking and crumbling between the fingers. This strength is a measure of the character and quantity of the colloidal fraction contained in the soil. The dry strength increases with increasing plasticity.

High dry strength is characteristic for clays of the CH group. A typical inorganic silt possesses only very slight dry strength. Silty fine sands and silts have about the same slight dry strength, but can be distinguished by the feel when powdering the dried specimen. Fine sand feels gritty whereas a typical silt has the smooth feel of flour.

TOUGHNESS (Consistency near plastic limit)

Earth Manual, A Water Resources Technical Publication, Second Edition, U. S. Department of the Interior, United States Government Printing Office Washington 1974, Reprinted in 1980

Mold a small sample to the consistency of putty adding water if necessary. If the sample becomes too sticky, spread the sample out in a thin layer and allow some of the soil moisture to evaporate. Roll the specimen out by hand on a smooth surface or between the palms into a thread about one-eighth inch in diameter. The thread is then folded and rerolled repeatedly. During this manipulation, the moisture content is gradually reduced and the specimen stiffens, finally loses its plasticity, and crumbles when the plastic limit is reached.

After the thread crumbles, the pieces are lumped together and a slight kneading action continued until the lump crumbles.

The tougher the thread near the plastic limit and the stiffer the lump when it finally crumbles, the more potent is the colloidal clay fraction in the soil and the higher is the plasticity. Weakness of the thread at the plastic limit indicates either inorganic clay of low plasticity, or materials such as Kaolin-type clay and organic clays.

Highly organic clays have a very weak and spongy feel at the plastic limit.

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Split-Spoon Sampling

SPLIT-SPOON SAMPLING

Soil borings that are drilled for a geotechnical study (i.e., a study designed to determine the compressive strength of the soil for the purpose of new building construction) are usually sampled at 2.5-foot intervals in the first 10 feet below grade and at 5-foot intervals thereafter to the bottom of the boring.

The depths from which soil samples are collected in an environmental investigation can be very site-specific. Soil samples are often collected from depths most likely to show environmental impact based on an evaluation of the known or suspected contaminants, the characteristics of the soils, and other variables that may affect a particular site.

Collecting a soil sample with a split-spoon or split-barrel sampling device is a common technique used to determine the physical soil characteristics and soil quality. It is described by ASTM Method D-1586 and is summarized below.

JOB DESCRIPTION:

Obtain soil samples at specified intervals, and collect the soil for laboratory analyses.

TASK-SPECIFIC EQUIPMENT NEEDED:

- · drilling and sampling devices
- tape for locating
- well/boring log sheets

EXPECTATIONS:

A well/boring log sheet will be accurately completed at each well or boring, according to Horizon's standard operating procedure "Well/Boring Log Guidelines", including blow counts, PID or FID response, soil descriptions, and other soil boring details.

The boring will be located by measurements and labeled on a site sketch or base map.

PROCEDURE:

- 1. Advance the boring to the desired sampling depth.
- 2. Attach the split-spoon sampling device to the bottom end of the drilling rods and gently lower it to the bottom of the borehole.
- 3. A 140-pound hammer free-falling a distance of 30 inches is used to drive the 2-inch O.D. split spoon 18 inches into the undisturbed soil below. Drive the 2-inch O.D. split-spoon sampler into the undisturbed soil ahead of the lead auger.
- 4. Record the number of blows required to drive the sampler for each 6-inch increment. If the soil is particularly hard and the blow counts are in excess of 100 blows per 6 inches, a split spoon may not be capable of obtaining the sample. Stop to keep from damaging the sampling device.
- 5. Bring the split spoon back to the ground surface after it has been driven over the sample interval.
- 6. Open the split spoon.
- 7. Field screen the sample if it is required.
- 8. Take samples that are collected for lab analyses from the mid to lower portions of the split spoon. Immediately place the soil that is most likely to be impacted (based on PID or FID response and visual observations of staining) into the appropriate sample bottles. Collect the samples to be analyzed for volatile organic compounds first. Collect the soil for semi-volatile analyses next, and collect soil for inorganic analyses last.
- 9. Place the samples in appropriate containers, using a clean tool and/or clean gloves.
- 10. Visually inspect the sample and describe it accurately and completely on the well/boring log sheet.

The upper portion of soil in the sampler can be disturbed or not representative of the sample interval targeted. This is because residual soils from within the auger stem may become entrained in the sample. The upper portion should be observed but field judgment should be used as to whether it is really representative of the sample interval. The upper portion should not be collected for lab analyses.

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Temporary Wells through Hollow-Stem Augers

TEMPORARY WELLS THROUGH HOLLOW-STEM AUGERS

Temporary wells are used to obtain a representative ground water sample from a discreet depth when permanent well construction is not desired.

PROCEDURE:

- 1. Advance the augers to the depth for the bottom of the well screen.
- 2. Remove the drill plug.
- 3. Lower the decontaminated well assembly to the bottom of the borehole.
- 4. If necessary, sand pack the well screen with clean, coarse sand grade silica sand to 1 foot above the top of the screen. Retract the augers while adding the sandpack.
- 5. Develop the well until sediment-free water is produced or at least three casing volumes are removed.
- 6. Sample the well with a clean bailer.
- 7. Pull the well out of the borehole.
- 8. Follow standard procedures for grouting the borehole.

Water Level Measurements

WATER LEVEL MEASUREMENT

There are two devices that are acceptable for measuring water levels. These are a steel tape and water-soluble carpenter's chalk, and an electric tape. While the electric tape reports measurements to 0.01 foot, it is less accurate than the steel tape method; therefore, the steel tape method should be preferred over the electric tape.

For some applications, the electric tape is preferred. An electric tape gives an accurate measurement to the water and is less likely to cross contaminate between wells that are to be sampled. Since the electric tape barely touches the water, it is easier to decontaminate after a water level measurement is taken. Water levels are obtained more quickly than with a steel tape.

The steel tape and chalk method is very accurate. This method, however, is a little less desirable for measuring water levels on wells that are to be sampled. The steel tape method introduces chalk to the water, the tape becomes rusted, and it is necessary to submerge a small portion of the tape to obtain a correct measurement.

The standard procedure for taking a water level measurement, whether using an electric tape or steel tape and chalk, is basically the same.

JOB DESCRIPTION:

Obtain a round of water levels.

TASK-SPECIFIC EQUIPMENT AND MINIMUM INFORMATION NEEDED:

- steel water level tape or electric water level indicator
- water-soluble carpenter's chalk
- paper towels
- hand-held engineer's measuring tape
- well location map
- well keys
- previous water level or water elevation data

EXPECTATIONS:

Water levels will be taken at all the indicated wells and recorded to the nearest 0.01 foot.

Document the time, date, and the method of the measurement.

PROCEDURES FOR A STEEL TAPE AND CHALK:

- 1. Uncap all the wells of a cluster to be measured to allow the water levels to stabilize. Be sure to place the well caps on a clean area (use visqueen if necessary). Vented well caps should provide for minimum (essentially zero) time for water level stabilization.
- 2. Smear the graduated portion at the end of the tape with chalk.
- 3. Carefully lower the tape into the well until the chalked portion of the tape intersects the water in the well.
- 4. Advance the tape until the nearest one-foot increment of the tape is exactly even with the top-of-casing; record.
- 5. Recoil the tape from the well and read the water level measurement directly from the wetted portion of the tape; record.
- 6. Dry the tape with a paper towel and resmear it with chalk, and take a second, confirmation measurement; record.
- 7. Clean the tape before proceeding to the next well.
- 8. Replace the well cap.

PROCEDURES FOR AN ELECTRIC WATER LEVEL INDICATOR:

- 1. Uncap the wells.
- 2. Carefully lower the tape into the well.
- 3. The buzzer will sound as the probe hits the water. Once the buzzer has sounded, slowly pull the tape up until the buzzer turns off.
- 4. Read the measurement from the top-of-casing and record it.
- 5. Take a second measurement to confirm; record it.
- 6. Clean the tape before proceeding to the next well.
- 7. Replace the well cap.

Each time a water level measurement is taken, a second confirmation reading is necessary to ensure that the water level is stable. If the second measurement is within ± 0.01 feet of the first, the measurement is good and can be recorded as a stable water level. If the second measurement does not confirm the first, then wait for the well to stabilize and try again.

Indicate in your field notes if the measurements were taken after or during a period of rainfall. Be alert to any irregularities observed which may have an effect on the water levels (such as a nearby pumping well).

Always record the date, time and method of each measurement.

If the measurement references a "holding point" other than the top of the casing, or you are unsure of which point is the top of casing, indicate the reference point used, measure the difference between the top-of-casing and the reference point, and provide a diagram.

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Well/Boring Log Guidelines

WELL/BORING LOG GUIDELINES

For every well or soil boring, a separate well/boring log sheet must be completed using standardized well/boring log sheet. On the top half of the sheet are a number of headings with accompanying blanks. It is imperative that all applicable information in this top area be filled out completely for each well or boring.

Well/Boring No.: The numbering sequence is generally provided by the project manager and must be recorded accordingly.

Client: Record the client or project name.

County-Township- This information can be filled out at the office by the project

Fraction-Section: geologist.

Contractor: Give the name of the contractor. Include their complete address. The equipment used should be documented. If a drill rig was used, record the make and the model. The name of the drilling crew chief should also appear here. The supervisor would be the person responsible for overseeing the work in the field.

Drilling Methods: Record the method of drilling that was used. Also, record the diameters of the drill string. Some examples would be:

- 4 1/4" I.D. HSA (inside diameter hollow stem auger)
- 10" O.D. HSA (outside diameter hollow stem auger)
- 3-1/8" mud rotary
- 5-1/2" O.D. x 3-1/4" I.D. dual wall reverse air circulation
- 4" O.D. hand auger

Grouting/Seal: Record the grouting material and the grouting method. If an additive is used, estimate its weight percentage. Also, record the bottom and top depths of the grout.

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Development: Include the developing method, rate, pumping time, and total volume evacuated from the well.

Screen: Each item needs to be completed as described on the sheet.

Casing: Record the casing material, diameter, and the bottom and top depths of the casing. Record the top of well thread distance to grade to the nearest 0.1 foot.

Date: Record as indicated on the sheet.

Elevation: Survey elevation data should be recorded here if you have this information. Be sure to include the reference point (whether it is U.S.G.S. datum or a relative elevation). Include the location of your reference point. This information may not always be available to you as the drilling takes place, and it may need to be filled in at a later date by others.

Water Level: In a soil boring, record the first water-saturated level encountered and the elapsed time before measurement. In a well, record the water level using the top of casing as the reference point and note TOC. Also, record the date of the measurement, the time elapsed since development, and the method of measurement.

Location: Reference your sketch and measurements to features evident on the base map which the project manager has supplied. If no base map has been supplied, reference your measurements to permanent site structures.

Well Sketch: Somewhere on the log sheet, a sketch of the well construction should be drawn. On this sketch, show the amount of above-ground stick-up, the depths to joints along the well casing, and the depth of the top and bottom of the well screen.

Remarks: Any added comments that are unique to the boring or well can be recorded here.

On the well/boring log sheet is an area for the lithologic description. The soils or rock that are penetrated during drilling should be recorded as accurately as possible according to the soils classification guidelines. Also on the log sheet, a number should be recorded representing the thickness and depth to base for every lithologic change recorded in the lithologic descriptions.

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Another area of the well/boring log sheet offers the opportunity to document soil sample depths, blow counts, and any other readings or measurements taken at individual depth intervals (Hnu, OVA).

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Well Casing Volume Calculation

WELL CASING VOLUME CALCULATION

Minimum information and equipment necessary to perform the task:

- well location map
- total depths of the wells
- water level tape
- calculator

Well casing volumes are important to determine the volume of water which must be purged from a well prior to collecting a groundwater sample which is representative of the screened aquifer. Obtaining this value is a two-step calculation:

 $V = "Pi" r^2H (7.48)$ is the equation for the volume of a cylinder and is used to make the volume calculation:

ONE:

where:

V = volume of water in the casing (cubic feet)

"Pi" = 3.14

r = radius of well (feet)

H = height of the water column in the well (feet)

7.48 = converts volume (V) from cubic feet to gallons

It is necessary to evacuate at least three volumes of water before sampling; therefore:

TWO: Vx3 = volume of water to be purged prior to sampling

A simplified form of the equation for the volume of the cylinder is:

$$V = r^2 H (0.163)$$

where:

V = volume of water in the casing (gallons)

r = the inside radius of the well¹ (inches)

H = the height of the water column in the well (feet)

 $H = H_0 - H_1$

 H_0 = total length of the well measured from TOC

 H_1 = the water level measured from TOC

0.163 = a constant

Carefully avoid the possibility of cross-contamination between wells by rinsing water level tape off between wells.

¹ If the field sampling plan requires that the filter pack volume be included, then the well radius used in the calculation should be the borehole radius. For hollow stem augers were used this should be roughly O.D./2.

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Field QC Sample Guidelines

FIELD OC SAMPLE GUIDELINES

The following is for guidance to staff who are making decisions in the work plan preparation phase of projects. This guidance applies to projects and clients that may not require field QC samples, but may benefit from the inclusion of them in the sampling plan as circumstances dictate.

With each new project, a conscious decision should be made whether to include QC samples. Agency requirements (e.g., the MDNR Waste Management Division Geotechnical Unit) may be specific for field QC samples. Therefore, be aware of agency requirements when responding to your client's ordered investigation.

TRIP BLANKS

Trip Blanks (organic free water samples in VOC vials placed in lab chest), are renewed each time a chest is packed or repacked with VOC sample containers. These samples remain unopened in the chest. If these "blanks" show "detectable" for one or more compounds, the problem could be cross-contamination between sample and container via air in the chest, or lab contamination.

Be sure these are packed with other volatile organic sample containers when five or more VOC samples will be taken that day, or when two or more sites will be visited on the same sampling trip. If possible, VOC sample containers should be packed and shipped in their own small cooler. VOC sample containers should definitely be separated from other sample containers that are visibly contaminated or "smell".

Analyze the trip blank for phthalates if base neutral compounds are to be run. These are contaminants that can enter samples from "plastic" lab containers and other sources. The laboratory provides trip blanks with every set of semi-volatile bottles.

Trip blank samples need not be analyzed routinely. But if a reported analysis is suspect, the blank can be run. Bear in mind that the value of a blank sample "on hold" decreases with time beyond one or two weeks due to possible communication by other samples via the lab air, and due to holding-time considerations.

FIELD BLANKS

Field Blanks are VOC vials filled in the field sampling area with organic-free water. This is done most often when the work site shows pid readings above the off-site background, or when local background readings can be established as non-zero.

Take one or more of these if the air screening instrument reads 2 ppm or higher above the background in the work area at any time during the day (not including sample-specific readings). This assumes instruments are calibrated in an off-site, clean-air area, where volatile contaminants are non-detect. The high air concentrations can be due to a persistent site condition, or to the presence of volatiles only at times of drilling and surfacing of contaminated subsoils or ground water. Field blanks should also be collected in conjunction with samples where there are vehicles or heavy equipment operating nearby, or when there is noticeable particulate matter.

Pack extra VOC vials, along with organic-free water in a non-reactive container. Obtain one or more blanks during the day by filling and sealing VOC vials with organic-free water while in the work zone. Leave the water container firmly closed otherwise. Analyze the blank taken at the time or closest to the time of the highest work-zone reading made during the day. Additional blanks may be run if conditions warrant.

The field blanks for sampling events involving water VOC samples, and for soil VOC samples prepared by adding organic-free water to a partially soil-filled vial, should be taken if site air readings are 2 ppm or more above ND background. For the usual method of collecting soils by completely filling the vial with soil and without adding water, the threshold may be 5 ppm or more above ND background.

EQUIPMENT RINSE BLANKS

Equipment Rinse Blanks are rinse water samples obtained after the final planned rinsing step for decontamination of bailers, split spoons, lead auger, etc. These blanks demonstrate that the non-dedicated sampling equipment has been thoroughly cleaned and that the sample collection and handling process has not altered the quality of the sample. These blanks typically include containers for all of the pollutant groups being analyzed. This kind of blank is most effective in demonstrating decontamination thoroughness when accompanied by a "before" rinse sample of organic-free water passed through the device immediately after using the equipment.

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A field-filtering blank should be collected when samples are filtered in the field.

UTILITY WATER SAMPLES

Utility Water (for drilling fluid make-up, wash/rinse water, etc.) samples should be taken for VOC analysis whenever a new or different source is used. If a source is used for which prior water quality knowledge is unavailable, it is a good idea to run all of the analytical groups being investigated.

Run one sample early in the investigation for all investigative groups unless existing data for the specific source valve indicate there is no need. If any compounds are detectable, rerun a sample of the final tank (drum) for those compounds. Alternatively, find an uncontaminated source of utility water.

DUPLICATE SAMPLES

Under any of the following conditions, obtain duplicate samples:

- 1. Each day seven or more investigative samples will be taken.
- 2. One duplicate for each ten samples during the day plus a duplicate sample beyond ten or a multiple.

Example:	0-6 samples during the day	0 duplicates
	7-10 samples during the day	1 duplicate
	11-20 samples during the day	2 duplicates
	21-30 samples during the day	3 duplicates

This means the analytical cost for duplicate samples where seven or more investigative samples are taken will add between 10 and 18 percent to the cost of the investigative samples. Lab discounts for multiple samples will help reduce the cost.

Duplicates may not be needed when a site will be sampled repeatedly, as with a quarterly monitoring sampling schedule.

SPECIAL CONSIDERATIONS FOR DUPLICATE SOIL SAMPLES

Obtain soil (or waste) duplicate samples at the same frequency as water samples. Because of the time required to perform some operations frequently involved with soil sampling-for example, obtaining a partial sample for later head-space screening; splitting the core and discarding "stones"; or describing a particularly detailed core-they can result in differential loss of VOC's from partial samples taken as duplicates. For this reason, volatile organic analyses from duplicate samples can disagree widely. To avoid this, care should be taken to handle duplicate VOC samples in the same manner as the investigative samples, especially the elapsed time between sample collection and sealing of the vials.

Additional problems with soil duplicates include the mass of soil needed for analysis (which sometimes exceeds the volume/mass in the sampler), and the difficulty of obtaining two separate split spoon samples at the same level for use as duplicates. Careful planning needs to be done when volatiles or a critical number of priority pollutant groups are to be analyzed.

The purpose of the duplicate samples should be clear before going to the field. If the purpose is to verify the sample handling and analytical procedures, then a split sample should be collected as a duplicate. If, however, the sampling methodology is to be verified, then a totally separate sample from a closely adjacent or offset location should be collected.

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Decontamination, Downhole Sampling Equipment

FIELD DECONTAMINATION, DOWNHOLE SAMPLING EQUIPMENT

This guideline consists of minimum requirements for the decontamination of split-spoon sampling devices, temporary well materials, augers, and water sampling pumps and bailers used at hazardous waste sites for obtaining samples to be analyzed in the laboratory.

The requirements apply foremost to equipment being used to obtain samples for the laboratory and, therefore, are intended to minimize incidents of cross-contamination of samples. The requirements also apply to the auger when contaminated soils are sampled only for on-site identification purposes. This is to avoid cross-contamination of locations particularly when moving from a "dirty" to a "clean" location.

ANALYTE TYPES

Different decontamination steps are recommended for different kinds and physical states of analytes. The analyte types for this purpose are:

- inorganics--major dissolved ions, along with heavy metals.
- dissolved organics--organics as aqueous species, no "free" organic phase present.
 - (1) soluble organics--ketones, alcohols, ethers, and others that are fairly to infinitely soluble in water
 - (2) VOC's--sparingly soluble
 - (3) semi-volatiles--sparingly soluble
 - (4) PCB's and pesticides--nearly insoluble in water, but very soluble in oil.
- free phase organics—a visible or suspected organic liquid phase, or "oil" from any of
 various sources. This is usually not an analyte, but when present interferes with
 analysis of the aqueous phase compounds and presents a strong potential for crosscontamination.

combination

The decontamination procedures apply to equipment in contact with analytes whether present within a soil matrix or as a "free" liquid.

DECONTAMINATION FREQUENCY

With a few exceptions to be mentioned, all named equipment should receive the same kind

of decontamination.

UPON ARRIVAL AT SITE

All augers and other equipment provided by the contractor should be decontaminated

upon arrival at the site. Equipment provided by Horizon which has not been pre-

decontaminated and suitably protected before and during transport to the site should also

be decontaminated upon arrival.

DURING SITE ACTIVITIES

All augers should be decontaminated between each location. The split-spoon sampler,

temporary well materials, and bailers should be decontaminated between each use. If

there is a delay between evacuation and sampling the well, the bailer should be

decontaminated before sampling. Pumps and bailers used for well development or

redevelopment should be decontaminated before using them for evacuation and sampling.

FIELD DECONTAMINATION PROCEDURES

The approved procedures for decontamination in the field are summarized in the

accompanying table.

SOLUBLE ORGANICS, VOC'S AND SEMI-VOLATILES

Tap water is typically used for steam cleaning. Steam cleaning should always be done at

"live steam" temperatures, which exceed 2120F. Be sure the steamer water is taken from

a public water supply or a source of known and approved quality. If you know or suspect

that unvaporized water is carrying over, halt work until the steamer is performing as it

should or use an alternative decontamination method. Also, be sure the steam delivery

wand is of sufficient length to deliver live steam to any remote points of the equipment.

INORGANICS AND HEAVY METALS

The inorganics and heavy metals call for an initial soapy wash, tap water rinse, and a final DI rinse. Excessive field concentrations of metals may make desirable a dilute nitric acid rinse prior to the DI rinse. The acid rinse need not be done routinely.

OTHER ORGANICS

The oils, PCB's, pesticides, and free organic phases call for a soapy wash and potable water rinse, steam, and a DI water rinse.

SPECIAL SITUATIONS

Certain situations may require a hexane solvent wash and 50% methanol-water solvent rinse in the field, such as PCB oils, coal tars, or motor oils in soils or as a LNAPL. Both solvents have low flashpoints, and normally would be delivered to the site by a DOT-licensed contractor.

The use of hexane should be preceded by a soap and water wash and followed by a 50% methanol-water rinse, steam cleaning and a DI rinse. The "soap" for the soapy wash can be a TSP (trisodium phosphate) product. However, if phosphate or phosphorus is an analyte, a low-phosphate detergent, such as Alconox, should be used.

Pump interior and conductor tubing decontamination, which could be ineffective or result in damage to equipment if steam is used, should follow the table procedure omitting the steam step. A 50% methanol-water solution, if available, may be substituted for the steam cleaning.

DECONTAMINATION UPON RETURN TO HORIZON PREMISES

All downhole equipment which is provided by Horizon that is not decontaminated in the field may be decontaminated in approved locations on Horizon premises upon returning from the field. Procedures are stepwise as follows for routine decontamination:

- Steam cleaning
- Alconox and water wash
- Steam cleaning
- Water wash

- Steam cleaning
- DI water rinse

Decontamination procedures for unusual or special situations should be substituted as described above under the FIELD heading.

Decontaminated bailers will be sealed into clean tubes until they are next used in the field.

ON-SITE STORAGE AND DISPOSAL OF DECONTAMINATION FLUIDS

Before storing fresh or spent methanol-water solutions, fuels, or hexane on site temporarily, assure that the site is secure (enclosed or patrolled) and that bermed containment or a large galvanized tub is provided for secondary containment.

In general, all spent wash and rinse waters including organic rinse liquids should be contained to prevent them from being returned to the ground. All decontamination liquids, along with other incidental waters such as well-development or well evacuation water, should be appropriately disposed. Disposal arrangements or plans should be developed prior to the work, where feasible, and with the client's input and concurrence. Appropriate disposal for liquids may include disposal to an on-site industrial wastewater treatment system.

"Spent" 50% methanol-water solution and spent decontamination water can often be discharged to a POTW via a sanitary sewer by prior arrangement. Because some analytical verification may be required, build some lead time into this request.

RECOMMENDED FREQUENCY OF CHANGING SOAPY WASH WATER AND RINSE WATER

The cleanest way to perform soap and tap water decon is to have a squirt bottle, sprayer or other applicator for the soapy water, and a flowing stream of tap water. In this manner, the soapy water and tap water "reservoirs" are always clean, and equipment comes in contact with clean solutions only.

If you have to use a tub or dip tank for immersion of equipment in soapy water, the following is offered. Related to the frequency of change is the order in which equipment should be decontaminated. Some general guidelines to minimizing the needed changes:

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- Wash cleaner (less soiled or "oily") equipment first.
- Have several split-spoon samplers on hand where practical, to save decontamination time and to allow washing these in a batch before the auger needs to be washed.
- Wash bailers in a dedicated soapy solution. This should not often be inconvenient
 since drilling/soil sampling is usually done separately from well sampling. Having
 several bailers on hand can allow batch washing of them before a dirtier piece of
 equipment needs to be washed.
- Wash temporary well casing and screens in a soapy solution dedicated to these materials only.

If a tap water stream is impractical, change the rinse water when it first shows discoloration, floating debris, or foaming tendencies due to soap carryover. Two rinse water baths in series, changing out both when the second bath reaches the above condition, will minimize changes.

Change soapy water when you change the rinse bath, sooner if the soapy water shows a slick that is other than detergent film. Change soapy water when the solution is too "spent" to generate soap/detergent foam with moderate agitation. If, before any of the above indications occurs, the soap solution becomes discolored with suspended clay or silt, it becomes a matter of personal judgment. It is a good idea to have sufficient drum capacity on hand to be able to change the soapy water "more" frequently rather than "less".

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Jar Head Space Measurements in Unsaturated Soil Samples JAR HEADSPACE MEASUREMENTS IN UNSATURATED SOIL SAMPLES

(USING FID OR PID)(1)

INTRODUCTION:

This procedure is most commonly used at sites where there is a suspected impact from gasoline constituents. The two instruments most commonly used for this field procedure are a flame ionization detector (FID) and a photoionization detector (PID). The FID response is uniform for most volatile gasoline hydrocarbons while the PID response increases for the BTEX compounds. Therefore, the PID may be more effective when concentrating on the aromatic constituents of gasoline.

Most field devices are sensitive to changing weather, and the response of the PID may become significantly affected by an increase in the humidity.

The FID systems, unlike the PID systems, will respond to methane.

GOAL:

To obtain a field estimate of the relative concentrations of total volatile organic compounds (VOC's) contained within a soil sample.

TASK-SPECIFIC EQUIPMENT NEEDED:

1. Flame ionization detector (FID) or a photoionization detector (PID) equipped with a 10.6 eV lamp.

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2. Glass sample jars between 9 and 16 ounces in total capacity.

3. Aluminum foil.

⁽¹⁾ John Fitzgerald, Petroleum Contaminants in Soil, Vol. II, pp. 119-135.

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PROCEDURE:

- 1. Calibrate the FID or the PID as indicated in the instrument manual.
- 2. Record the calibration procedure and the calibration results in the field notes. If a dedicated log book accompanies the instrument, record the calibration details in it.
- 3. Collect the soil sample.
- 4. Place the soil sample into the glass sample jar immediately. Fill the sample jar half-full.
- 5. Seal the sample jar by placing a clean piece of aluminum foil over the mouth and threads of the jar.
- 6. Allow the sample to reach approximately 70°F.
- 7. After a 5- to 10-minute headspace development period, vigorously agitate the sample jar for at least 30 seconds.
- 8. Immediately insert the probe of the FID or PID through the aluminum foil seal and into the sample jar.
- Record the maximum meter response as the TOTAL ORGANIC VAPOR HEADSPACE concentration on the Well/Boring log form or the field notes as appropriate.
- 10. Record any significant changes in the weather (and the apparent humidity) that occur throughout the day.

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STANDARD PHOTOIONIZATION DETECTOR (PID) OPERATIONS AND PROCEDURES

Introduction

The HNU Model PI 101 is designed to measure the concentration of trace gases in the atmosphere. The principle of photoionization detection (PID) is employed. A sensor, consisting of a sealed ultra-violet light source (either 9.5 eV, 10.2eV, or 11.7 eV) emits photons energetic enough to ionize many trace species of organic hydrocarbons. The ionized gases are in turn detected by a collector electrode where the current is measured and converted to a ppm value. The useful range of the instrument is from a 1 ppm to 2,000 ppm. This instrument is used frequently by WW Engineering & Science to evaluate ambient air quality for health and safety purposes and to detect the presence of volatile organic hydrocarbons in soil and sediment samples.

Operation

- 1. Turn the function switch to the "battery check" position. The needle on the meter should read within the green area (battery area) of the scale; if not, the battery should be recharged.
- 2. Turn the function switch to the "on" position. Look into the end of the probe and confirm the purple glow of the UV lamp.
- Zero the instrument; turn the function switch to the "standby" position and rotate the zero potentiometer until the meter reads zero. Clockwise rotation = upscale deflection; no calibration gas is necessary for this adjustment. Confirm zero rating is stable; if not, readjust.
- 4. Calibrate the instrument; turn the function switch to the proper measurement range (specific to the calibration gas). Connect the sensor to the provided cylinder of calibration gas, open the valve on the cylinder. Use the span control to adjust the instrument scale reading to the ppm value specified on the cylinder of calibration gas. The instrument is now ready for use. Be sure to position the function switch to "stand-by" between observations to prevent unnecessary drain on the battery.
- 5. To prevent the undetected escape of volatile vapors when scanning split-spoon soil or sediment cores, have the instrument at the ready when the split-spoon is opened. Immediately upon opening the corer or split-spoon, disturb the sample and scan representative areas of the sample.

- 6. The battery should be recharged each night. To charge the battery, place the miniphone plug into the jack prior to plugging in the 120 VAC. When disconnecting the charge, remove from the 120 VAC before removing the mini-phone plug. Check the battery to confirm its charge.
- 7. If the probe is held near AC power lines or transformers, an error may be observed. If AC "pick-up" is going to be a problem, the meter, in "stand-by" position, will indicate the magnitude of the error rather than reading zero. This may be taken into consideration and the error compensated for by simply subtracting the value observed when the instrument was on stand-by from the observed detected value when making a positive reading.
- 8. The HNU PID is affected by humidity. It will not function properly in rainy weather, and "negative" deflection and difficulty with zeroing the instrument can occur under otherwise humid conditions.

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Soil Boring Drilling using Hollow-Stem Augers

SOIL BORING DRILLING USING HOLLOW-STEM AUGERS

INTRODUCTION:

When the primary objective of the drilling is to obtain soil samples from discreet depths, the hollow-stem augering (HSA) technique of drilling is one of the most effective. The soil is penetrated with five-foot-long, continuous helical flight augers which are driven by a rotary drive head mounted on a hydraulic feed system which pushes the drill stem down or pulls it up. Cuttings are mechanically removed from the borehole by the flights on the HSA's.

GOAL:

To drill a soil boring from which the depths and descriptions of the soils encountered can be accurately logged and to obtain samples of the soils from accurate soil depth intervals.

TASK-SPECIFIC EQUIPMENT NEEDED:

All drilling equipment and labor are supplied by the subcontracted drilling company.

PROCEDURE:

- 1. Access the drill rig and all necessary equipment to the proposed borehole location.
- 2. Advance the HSA's to the top of the proposed soil sampling depth.
- 3. With the augers in place and at rest, remove the center plug from the inside of the augers.
- 4. Attach a decontaminated split-spoon sampling or other device to the drilling rods.
- 5. Lower the device inside the HSA to the bottom of the borehole.
- 6. Drive the device into the soil as described, for example, in the standard operating procedure (SOP) for "Split-Spoon Sampling."
- 7. Recover the device.
- 8. Replace the plug inside the HSA and continue drilling to the next sampling depth.

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9. When the desired completion depth has been attained, properly backfill the borehole from the bottom up as described in the SOP for "Soil Boring Abandonment" and decontaminate the drilling and sampling tools according to the SOP "Field Decontamination, Downhole Sampling Equipment."

Unless there is a specific need for another size auger, standard procedure is to drill soil borings with 4.25-inch inside diameter HSA's. The outside diameter of these augers is 8.25 inches.

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Packing and Shipping Samples for Laboratory
Analysis

PROCEDURES FOR PACKING AND SHIPPING SAMPLES

FOR LABORATORY ANALYSIS

GENERAL:

Sample packaging and shipping procedures are based on U.S. EPA specifications as well as Department of Transportation (DOT) regulations (49 CFR). The procedures vary according to sample concentration and matrix and are designed to provide optimum protection of samples and the public.

All samples are to be shipped via Federal Express, Purolator, or Emery as specified in the U.S. EPA Region V Sample Handling Protocol for Hazardous Waste. Shipping containers must be insulated, durable and water tight. Bagged samples are to be cushioned within the shipping container with absorbent packing materials to prevent breakage and leakage (vermiculite, zonolite, bubble pack or similar materials).

SAMPLE QUALITATIVE CONCENTRATION DEFINITIONS:

Low concentration organic samples are those containing less than 10 ppm of any of the "priority pollutants" including antimony, arsenic, beryllium, cadmium, chromium, copper, lead, mercury, nickel, selenium, silver, thallium, zinc, and cyanide. Medium concentration inorganic samples are those containing between 10 ppm and 15 percent of any of the priority pollutants. High concentration inorganic samples are those containing above 15 percent of any of the priority pollutants. Concentrations of the samples can be estimated by using existing data, if available.

Step by step packing instructions are provided below.

LOW CONCENTRATION SAMPLES:

- 1. Prepare cooler(s) for shipment.
- Tape drain shut.
- Affix "this Side Up" labels on all four sides and "Fragile" labels on at least two sides of each cooler.

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- Place mailing label with laboratory address on top of cooler(s).
- Fill bottom of cooler(s) with about three (3) inches of vermiculite.
- Place appropriate chain-of-custody (COC) records on top of each cooler.
- 2. Arrange decontaminated sample containers in groups by sample number.
- 3. Mark volume levels on bottles with a grease pencil.
- 4. Secure appropriate sample tags around caps / lids of containers with string or wire.
- 5. Arrange containers in front of assigned cooler(s).
- 6. Arrange containers in coolers so that they do not touch.
- 7. If ice is required to preserve the sample, cubes should be repackaged in double zip-loc bags and placed on and around the containers (especially on VOC vials).
- 8. Fill remaining space with absorbent material to prevent breakage.
- 9. Sign chain-of-custody form and indicate the time and date it was relinquished to Federal Express, Purolator, or Emery.
- 10. Separate the copies of COC forms. Seal proper copies within a large zip-loc bag and tape to inside lid of cooler. If you are not returning to the office within the week, place remaining copies in a large mailing envelope to be sent to the Horizon project manager.
- 11. Close lid and latch.
- 12. Tape cooler shut on both ends, making several complete revolutions with strapping tape (do not cover custody seals).
- 13. Relinquish to Federal Express. If you are not returning to the office within the week, place airbill receipt inside the mailing envelope and send to the Horizon project manager along with the other documentation (Item 10).

MEDIUM AND HIGH CONCENTRATION SAMPLES

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Medium and high hazard samples shipped by Horizon Environmental Corporation personnel are subject to DOT regulations. Therefore, to comply with the prescribed regulations, all Horizon personnel must abide by the following procedures.

- 1. Collect samples in appropriate containers as required. Assure that the sample container cap is sealed with tape.
- 2. Attach sample tags to each sample as required.
- 3. Place each sample in a zip-loc bag in such a way that the sample tag can be read.
- 4. Place each sealed bag inside a metal can and fill the can with absorbent cushioning material such as vermiculite. The can must be sealed, preferably using clips but tape may also be used.
- 5. Place the name and address of the laboratory on the can.
- 6. Place a "Flammable Liquid, n.o.s." or "Flammable Solid, n.o.s." label on the can.
- 7. Place a "cargo Aircraft Only" label on the can.
- 8. Place each can in the shipping container (cooler) which has been lined with two (2) inches of absorbent material.
- 9. Surround each can with absorbent material to prevent sample breakage and provide stability during transport; fill the shipping container with absorbent material.
- 10. Place all shipping paperwork to accompany the samples, excluding the airbill and Shipper's Declaration of Dangerous Goods, in a manila envelope. Secure the envelope in a zip-loc bag and place the bag on top of the absorbent material in the cooler.
- 11. Close and seal the cooler using strapping tape.
- 12. Mark the shipping container with the following information and labels:
- Shipping address.
- Laboratory address (consignee).

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- "This End Up", with arrow, label (for liquids only).
- "Cargo Only Aircraft" label.
- "Inside Containers Comply with Prescribed Regulations" label.
- "Flammable Liquid" or "Flammable Solid" label.
- Additional hand-written label indicating DOT proper shipping name, e.g., "Flammable Liquid, n.o.s. UN1993, or "Flammable Solid, n.o.s. US 1325" (This is required only if the "Flammable Liquid" or "Flammable Solid" labels do not exhibit the applicable DOT proper shipping name). These labels need only be placed on one face of a cooler. Packages having a volume greater than 64 cu. ft. (4'x 4'x 4') require labeling on two (2) sides or ends.
- 13. To ship packaged samples, the samplers need only to fill out an airbill and for medium and high hazard samples, a Declaration of Dangerous Goods.
- 14. Same as step #13 for low concentration.

Individual Federal Express offices may have different preferences for completing the Dangerous Goods form. Prior to sampling, it is recommended that you contact the office from which you are shipping and also call the Federal Express 1-800 number (on the airbill) and ask for their "Hazardous Materials shipping section".

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Field Determination of pH

FIELD DETERMINATION OF PH

GROUND WATER, SURFACE WATER AND LEACHATE ANALYSIS

Method Summary

This is a determination of the activity of the hydrogen ions by potentiometric measurement.

Interferences

Temperature is an important factor. The temperature compensator attached to the instrument automatically corrects the pH value displayed by the meter.

Instrumentation

Beckman pH meter pH probe Automatic Temperature Compensator (ATC)

Materials and Reagents

Sample cups Prepared pH 4 and 10 standards for calibration

Calibration

During initial setup and calibration, two standards are run.

Standardizing the Instrument

- 1. Depress the CLEAR key to clear the instrument.
- 2. Rinse the electrode with distilled water and immerse in pH 4 buffer. Depress the STANDARD key. When the input from the electrode is stable, the instrument will automatically standardize on the pH value of 4.00 pH buffer. The STD1 symbol and the approximate value of the pH 4.00 buffer will appear in the DISPLAY.
- 3. Rinse the electrode with distilled water and immerse in pH 10 buffer. Depress the STANDARD key again. When the instrument stabilizes, the DISPLAY will include STD1, STD2, temperature and the approximate value of the pH buffer 10.

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- 4. The instrument is now ready to make a pH measurement. Rinse the electrode with distilled water and immerse in the sample.
- 5. Depress the pH key. Wait until the AUTO symbol flashes and then locks. The DISPLAY will indicate the measured temperature and pH.
- 6. This sequence can be repeated for additional pH measurements. Depress the pH key, wait for AUTO READ to lock, and note the pH value.
- 7. A +0.05 pH acceptance limit should be used in determining calibration acceptability. If unacceptable, recalibrate.

Measurement Procedure

- 1. Prepare and analyze samples without delay.
- 2. Place about 50 mls of sample into a plastic cup and stir with the pH probe.
- 3. Allow the pH reading to stabilize. Collect three pH readings from each sample within ± 0.5 units. Record the pH values on the well or surface water sampling record form. Rinse the probe with distilled water and verify calibration by submersing in a prepared pH standard as described under "Calibration".
- 4. Proceed to the next sample or location; verify calibration before each measurement.

Quality Control

- 1. Document all calibrations and verification readings, including time and meter readings.
- 2. Run duplicate measurements on each batch or every 10th sample.

Maintenance

- 1. Check battery (if used in field); and replace if discharged.
- 2. After use in samples containing free oil, wash the electrode in soap and rinse thoroughly with water. Immerse the lower third of the electrode in diluted HCL (1:9) solution for 10 minutes to remove any film formed. Rinse thoroughly with water.
- 3. Keep electrode properly filled with appropriate filling electrolyte solution.

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Field Determination of Conductivity, Method 205

FIELD DETERMINATION OF CONDUCTIVITY, METHOD 205

GROUND WATER, SURFACE WATER AND WASTEWATER

Method Summary

Conductivity is a numerical expression of an aqueous solution's ability to carry an electric current. This is dependent on the presence of ions, their concentrations, mobility, valence, and on the temperature of the solution.

The conductivity probe is immersed in a sample and the conductivity is read directly off of the meter scale.

Interferences

Temperature greatly influences the electrolytic conductivity of a sample, therefore, it is extremely important accurate temperature measurements are made.

Instrumentation

Conductance meter YSI Model 32.

Materials and Reagents

Conductivity cell

Thermometer

Specimen containers

Standard

Primary Working Standard:

Potassium chloride standard 0.01N: dissolved 0.7456 g anhydrous KCII in deionized water and dilute to 1 liter at 25°C. Conductivity = 1,413 umhos/cm.

Calibration

Check the conductivity of the standard prior to actual sample evaluation. Record the temperature of each standard.

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Calculate the conductivity at 25°C making adjustments for the temperature (see "Calculations", below).

Procedure

- 1. Rinse the cell with deionized water.
- 2. Measure the conductivity of each sample by swirling the cell in a portion of the sample. Record the conductivity reading and the temperature. Collect three conductivity readings until the readings are within ±5 umhos/cm.
- 3. Calculate the conductivity at 25°C as outlined in "Calculations", below.

Calculations

Conductivity at 25°C =
$$\frac{K}{1 + 0.0191(t-25)}$$

K = measured conductivity t = temperature of sample, °C

Quality Control

- Document all calibrations and verification of readings including time and meter readings.
- A blank of deionized water is run and should have a conductivity of less than 5 umhos/cm.
- The initial standard is checked in between samples.
- Duplicate measurements of conductivity will be taken at least once for every 10 investigative samples.

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Soil and Ground Water Sampling Using the Geoprobe

SOIL AND GROUND WATER SAMPLING USING THE GEOPROBE

The geoprobe system of samplers and tools is used for the collection of discreet soil and ground water samples. This system incorporates stainless steel sampling tubes with disposable liners and mechanisms for sample collection at specific depths with the intent to collect soil profile samples with minimal disturbance of the existing conditions and small diameter screens or slotted pipe for the collection of soil vapor or ground water samples.

Several different methods are used to advance the sampling tools to depth. The method used is often dependent on accessibility to the sample location and type of materials being sampled. Sampler advancement can be by impact hammer, hydraulic force or hand driven methods. Sampler extraction most often is by hydraulic force.

LIMITATIONS

Specific site conditions can also limit this method of sampling with the presence of rubble and debris and equipment accessibility problems. Because this method introduces the sampler through the same uncased hole for each sample interval the potential for cross-contamination must be considered.

SOIL SAMPLE COLLECTION AND HANDLING PROCEDURE

All sampling equipment are properly decontaminated before sample collection begins. Samplers incorporate a disposable liner to assist in sample handling and reduce sampler decontamination. Sampler liners are available in several different material composition.

Large Bore Sampler: A 24-inch long x 1-3/8-inch diameter piston-type soil sampler capable of recovering a discrete sample that measures up to 320 ml in volume, in the form of a 22-inch x 1-1/16-inch core contained inside a removable liner.

Liner: A 24-inch long x 1-1/8-inch diameter removable/replaceable, thin-walled inserted inside the Large Bore Sampler body for the purpose of containing and storing soil samples. Liner materials include brass, stainless steel, Teflon, and clear plastic (either PETG or cellulose acetate butyrate).

The assembled Large Bore Sampler is connected to the leading end of a Geoprobe brand probe rod and driven into the subsurface using appropriate methods. Additional probe rods are connected in succession to advance the sampler to depth. The sampler remains

sealed (closed) by a piston tip as it is being driven. The piston is held in place by a reverse-threaded stop-pin at the trailing end of the sampler. When the sampler tip has reached the top of the desired sampling interval, a series of extension rods, sufficient to reach depth, are coupled together and lowered down the inside diameter of the probe rods. The extension rods are then rotated clock-wise (using a handle). The male threads on the leading end of the extension rods engage the female threads on the top end of the stop-pin, and the pin is removed. After the extension rods and stop-pin have been removed, the tool string is advanced an additional 24 inches. The piston is displaced inside the sampler body by the soil as the sample is cut. To recover the sample, the sampler is recovered from the hole and the liner containing the soil sample is removed.

Pilot Hole

A pilot hole is appropriate when the surface to be penetrated contains gravel, asphalt, hard sands, or rubble. Pre-probing can prevent unnecessary wear on the sampling tools. A Large Bore Pre-Probe may be used for this purpose. The pilot hole should be made only to a depth above the sampling interval. Where surface pavement is present, a hole may be drilled with the Geoprobe using a drill steel with a 1.5-inch diameter carbide drill bit prior to probing. For pavements in excess of 6 inches, the use of compressed air to remove cuttings is recommended.

Sample Collection

- 1. When sampling depth has been reached, position the drive equipment away from the top of the prove rod to allow room to work.
- 2. Insert an AT-67 Extension Rod down the inside diameter of the probe rods. Hold onto it and place an AT-68 Extension Rod Coupler on the top threads of the extension rod (the down-hole end of the leading extension rod should remain uncovered). Attach another extension rod to the coupler and lower the jointed rods down-hole.
- 3. Couple additional extension rods together in the same fashion as in Step 2. Use the same number of extension rods as there are probe rods in the ground. The leading extension rod must reach the stop-pin at the top of the sampler assembly. When coupling extension rods together, you may opt to use the GW-469 Extension Rod Jig to hold the down-hole extension rods while adding additional rods.

- 4. When the leading extension rod has reached the stop-pin down hole, attach the AT-69 Extension Rod Handle to the top extension rod.
- 5. Turn the handle clockwise (right-handed) until the stop-pin detaches from the threads on the drive head. Pull up lightly on the extension rods during this procedure to check thread engagement.
- Remove the extension rods and uncouple the sections as each joint is pulled from the hole. The Extension Rod Jig may be used to hold the rod couplers in place as the top extension rods are removed.
- 7. The stop-pin should be attached to the bottom of the last extension rod upon removal. Inspect it for damage. Once the stop-pin has been removed, the sampler is ready to be re-driven to collect a sample.
- 8. Reposition the Geoprobe Drawing equipment over the probe rods, adding an additional probe rod to the tool string if necessary. Make a mark on the probe rod 24 inches above ground surface (this is the distance the tool string will be advanced).
- 9. Attach a drive cap to the prove rod and drive the tool string and sampler another 24 inches. Do not over-drive the sampler.
- 10. Remove the drive cap on the top prove rod and attach an AT-12B Cap
- 11. Sampler retrieval can be by hydraulic force incorporating a jacking device or by methods like the Geoprobe vehicle-mounted machine that is designed to both tow and retrieve sampling equipment.

Sample Recovery

- 1. Detach the 2-foot probe rod it is has not been done previously.
- 2. Unscrew the cutting shoe using the At-669 LB Cutting Shoe Wrench, if necessary. Pull the cutting shoe out with the liner attached. If the liner doesn't slide out readily with the cutting shoe, take off the drive head and push down on the side wall of the liner. The liner and sample should slide out easily.

- 3. The ends of the liners can be capped off using the AT-641 Vinyl End Cap for further storage or transportation. A black end cap should be used at the bottom (down end) of the sample core and a red end cap at the top (up end) of the core.
- 4. On brass, stainless steel, and teflon liners, cover the end of the sample tube with At 640T Teflon Tape before placing the end caps on the liner. The tape should be smoothed out and pressed over the end of the soil core so as to minimize headspace. However, care should be taken not to stretch and, therefore, thin the teflon tape.
- 5. Large Bore Clear Plastic and Teflon Liners can be slit open easily with a utility knife for the samples to be analyzed or placed in appropriate containers.
- 6. Large Bore Brass and Stainless steel liners separate into four 6-inch sections. The AT-659K Large Bore Manual Extruder may be used to push the soil cores out of the liner sections for analysis or for transfer to other containers.

Decontamination

Sampling equipment decontamination can be one or a combination of soapy water wash and clean water rinse; steam cleaned, or a solvent wash and clean water rinse, dependent on analytical and cross-contamination concerns.

GROUND WATER SAMPLING PROCEDURE

All sampling equipment will be properly decontaminated before sample collection begins. The objective of this procedure is to drive a sealed stainless steel screen to depth, open the screen, and obtain a water sample via a tubing system to the surface.

Screen Point Ground Water Sampler: The assembled Screen Point Sampler (P/N GW-440K) is 1.0 inch O.D. (outside diameter) x 36-inch overall length. This sampler features a 19-inch screen encased in a perforated stainless steel sleeve. The device is also useful for measurement of piezometric levels.

The assembled Screen Point Sampler threads onto the leading end of a Geoprobe probe rod and is driven into the subsurface using appropriate methods. Additional probe rods are connected in succession to advance the sampler to depth. While the Screen Point Sampler is being driven to the desired sampling depth, it is kept sealed by O-ring connections placed at critical locations on the assembly.

When the desired sampling depth is reached, the sampler is pulled up about 2 feet which disengages the expendable drive point and creates an open borehole from which to sample. The inner core, which consists of a stainless steel sire screen inside of a perforated stainless steel sleeve, is then pushed out into the borehole and water is allowed to enter the sampler and connected probe rods.

In common practice, ground water samples are recovered by pumping or bailing of water collected in the open probe rods. Alternately, tubing from the surface may be connected directly to the sampler screen using a Geoprobe PR (post run) fitting, and samples recovered using a peristaltic pump or vacuum source. The pore size of the screen of this sampler is .0057 inches (0.145 mm). This sampler will allow the user to collect relatively clean water samples in a short time period due to its large surface area.

Sampler Installation

- Drive the water sampler approximately two-foot below the depth level where you want to sample by simply attaching it to Geoprobe rods.
- Never drive the water sampler without the O-ring (P.N GW-445R) attached to the drive point. Failure to use this O-ring may result in flowing soils to clogging the screen during driving.
- Retract the probe rods from the ground a distance of 24 inches (607 mm).
- Insert Geoprobe stainless steel extension rods (P/N AT-67) down the bore of the probe rods. An extension rod coupler (P/N AT-68) must be placed at the bottom end of the lead extension rod in order to protect the threads at the end of this rod. One extension rod will be required for each probe rod in the ground, plus one extension rod for the screen point sampler itself. Place an extension rod handle (P/N AT-69) at the top of the extension rod string.
- When the proper number of extension rods have been coupled together and inserted down the bore of the probe rods, the last extension rod will protrude from the top of the probe rods a distance of approximately 24 inches (607mm).
- Pushing down ton the extension rods should now push the screen out into the formation. When the screen is completely pushed out, the extension rod handle will

come to rest at a final position approximately 3 inches (76 mm) above the top of the probe rods.

 In extreme situations, it may be necessary to tap on the top of the extension rod handle with a hammer in order to force the screen out into the formation.

Ground Water Sample Collection

There are two methods for obtaining a sample from the GW-440 series Screen Point Sampler. Ground water samples can be obtained by bailing or pumping directly from the bore of the probe rods above the screen point. Alternately, a tubing system may be attached directly to the top of the deployed screen and samples pumped to the surface using either a peristaltic pump or other means of vacuum lift.

Bottom Check Valve Sampling

The most common methods employed is to pump directly from the bore of the probe rods immediately above the screen point using a tubing bottom check valve. This method is often referred to as sampling from the open rods, and is essentially the same for bottom check valve sampling as it is for bailing. Note that in order for this method to be employed, the piezometric head in the saturated formation must be above the top of the deployed screen point; water from the formation must rise into the probe rods where it can then be pumped to the surface. Sampling is performed as described in the following steps.

- Either 3/8 (9.5 mm) O.D. Teflon (P/N TB-30T) or Polyethylene (P/N TB-25L) tubing may be used for ground water sampling. Selection of tubing material should be based on the analytes of interest and the purpose of the ground water investigation.
- Place a tubing check valve (P.N GW-42) at the bottom end of a roll of tubing (fig. 4.12). This bottom check valve will fit either of the tubing types listed above.
- Push the tubing, check valve end first, down the bore of the probe rods until it strikes the top of the screen point sampler.
- Lift the tubing approximately 4 inches *(102 mm) off the bottom (top of the screen point sampler) and oscillate the tubing up and down in 8 to 12 inch (200 to 300 mm) strokes. In field practice, the tubing is oscillated up and down by hand at a rate of 60

to 100 strokes per minute. This pumping can yield as much as 500 milliliters of sampler per minute.

- Air bubbles appearing in the pumped stream indicate that the pumping action is
 exceeding recharge from the screen point, allowing air to enter at the check valve end.
 For most purposed, intermixing of air with the pumped sample is undesirable. The
 pumping rate should be slowed and balanced with the recharge rate.
- If water cannot be pumped to the surface, sufficient sample may be obtained by using the tubing and check valve as a bailer. Oscillate the tubing to fill it with several feet of sample and then remove the tubing from the rods.

Sampling Through PRT

"PRT" (post run tubing) refers to a Geoprobe proprietary system of tubing and fittings that are used both for vapor and ground water sampling. This tubing is inserted down the rods after the sampler has already been driven to depth and has been deployed for sampling. The top of the screen point sampler screen is equipped with a PRT fitting which serves as a receptacle for a corresponding PRT adapter fitted onto the end of the sample tubing.

In practice, the tubing with PRT adapter at the lower end is inserted down the bore of the probe rods and screwed into the receptacle on the top of the sampler screen. This procedure forms a vacuum tight sample train from the sampler screen to ground surface. Sample is normally pumped to the surface using a peristaltic pump or other vacuum source.

The advantage of this method is that the sample is only placed in contact with the stainless steel sampler screen and the sample tubing. The sample is never exposed to a free surface. The disadvantage of this method is that it is limited to maximum ground water depths of 20 to 28 feet (6 to 8.5m) below ground surface.

The following procedures are used to obtain ground water samples using PRT fittings and tubing:

• Either 3/8 inch (9.5 mm) O.D. Teflon (P/N TB-30T) or Polyethylene (P/N TB-25L) tubing may be used for ground water sampling. Selection of tubing material should be based on the analytes of interest and the purpose of the ground water investigation. Each of these tubing's has a corresponding PRT adapter that will be required for this sampling. These adapters are shown in the following table.

TUBING AND PRT ADAPTERS

Tubing	<u>Description</u>	PRT Adapter Part Number
TB-30T	3/8 inch (9/5 mm) TFE	PR-30S
TB-25L	3/8 inch (9.5 mm) LDPE	PR-25S

- Place the barbed end of the appropriate adapter into the selected tubing.
- Push the adapter end of the tubing down the bore of the probe rods until it comes into contact with the PRT threads at the top of the screen point sampler.
- Rotate the tubing counter-clockwise at the surface to screw the adapter in to the screen point threads. Rotate the tubing several revolutions until the down hold adapter is completely seated and the tubing starts twisting. In this condition, the tubing will rotate backwards (clockwise) when released.
- The tubing can now be attached to a peristaltic pump or vacuum source at the surface.
- After sampling is complete, tubing should be removed by pulling it up at the surface.
 This will pull the tubing off the barbed end of the tubing adapter and will allow the
 operator to examine the connection at the top end of the screen point when it is
 pulled from the ground.

Sampler Removal

- Remove all sampling tubing from the bore of the probe rods.
- Pull all probe rods from the ground using the extraction equipment. Care should be taken not to push down on the probe rods during removal.

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- Care should be taken to lift the screen point sampler vertically upward at the surface. Pulling the probe rods or sampler from the ground at any direction other than vertical may result in bending of the screen point sampler.
- Dismantle the sampler at the surface and examine if for damage. Decontaminate all parts, replace all O-rings, and reassemble the sampler for the next sample.

Decontamination

In order to assemble the water sampler properly and to take accurate and precise water samples, all parts need to be cleaned thoroughly and, if necessary, individually decontaminated prior to their use. For each test run, fresh, decontaminated sampler parts and O-rings should be used.

All parts should be washed with soapy water. All soil adhering to the parts should be removed by brushing or pressure washing. Finally, all parts should be rinsed with clean, contaminant-free water and allowed to dry before they are assembled.

Check all five O-rings in the sampler assembly for damage and/or wear. For reliable tests, we recommend the use of new O-rings on this tool at each sampling. It is more efficient and cost effective to change O-rings rather than collecting a non-representative sample or invalid data.

Appendix C

Field Forms

Well/Boring Log Sheet

Water Level and Field Record Form

Ground Water Sampling Field Record Form

Sample Tag

Chain-Of-Custody Record

Internal Field Audit Checklist

Well/Boring Log Sheet
Water Level and Field Record Form
Ground Water Sampling Field Record Form
Sample Tag
Chain-Of-Custody Record
Internal Field Audit Checklist

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 $[\]star$ = The USCS symbol assigned is based on visual and manual observations and not on tests performed in the laboratory.

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Water Level and Field Record Form

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Groundwater Sampling Field Record Form

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Project No:	
Date:	

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Analytical Services



EARTH TECH

5555 Glenwood Hills Parkway SE PO Box 874 Grand Rapids MI 49588-0874

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WW Engineering & Science A Summit Company

Chain of Custody Record

COC No.

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A Summit Company

5555 Clerrwood Hills Pkwy SE • PO Box 874 • Grand Rapids, MI 40588-0874

Analytical Services

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^{*} Matrix: Water (WTR), Wastewater (WW), Soil (SOIL), Sludge (SLG), Air, Oil, Waste (WASTE)

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APPENDIX C

INTERNAL FIELD AUDIT CHECKLIST

Name of Inspectors:	
Date of Inspection:	
1. REVIEW THE FIELD LOGBOOK RECORDS.	
Is each field logbook a bound field survey book or notebook?	
Is each logbook stored in the document control center when not in use?	
Is each logbook identified by the project-specific document number?	
Does each logbook contain a title page with the following information: Person to whom the logbook is assigned? Logbook number? Project name? Project start date? Project end date?	
Does each daily entry in the logbook begin with the following information: Date? Start time? Weather? Names of all sampling team members present? Level of personal protection used? Signature of person making the entry?	
Are the names of visitors to the site, field sampling or investigation team person purpose of their visit entered into the logbook?	nnel and the
Are all measurements entered into the logbook?	
Are all collected samples entered into the logbook?	
Are all entries made in ink?	
Are any erasures present?	
Are incorrect entries crossed out with a single strike mark?	
Is a detailed description of the location of the station which was sampled or me compass and distance measurements noted in the logbook?	asured including
Are the number of photographs taken, if any, recorded?	
Is all equipment used to make measurements, along with the date of calibration	ı, recorded?

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Is all equipment used to collect samples recorded?

Is the time of sampling recorded?

Is the sample description recorded?

Is the depth at which the sample was collected recorded?

Is the volume and number of containers recorded?

2. REVIEW THE CHAIN-OF-CUSTODYS

Are all samples accompanied by a properly completed Chain-of-Custody form, including sample numbers and locations, signatures, date and time of transfer?

Were the pink and yellow copies of the Chain-of-Custody form retained by the sampler and returned to the sampling office?

3. OBSERVE THE SAMPLE SHIPMENT

Are samples packaged in baggies and surrounded by vermiculite or bubble pack?

Are they dispatched to the appropriate laboratory?

Are they being sent by overnight carrier?

Is an original, signed Chain-of-Custody form enclosed with each shipment?

Is each shipping container locked and secured with strapping tape and custody seals in at least two locations?

Are the custody seals covered with clear plastic tape?

Were receipts of the bills of lading retained as part of the permanent documentation?

4. SAMPLE CONTAINER PREPARATION AND PRESERVATION (see Table 4.1 of the FSP)

Were the samples placed into the proper container?

Were they properly preserved?

5. DECONTAMINATION PROCEDURES

Were all tools which came into contact with potentially contaminated water, soil, or sediment decontaminated after each use?

Were the tools washed in an alconox solution?

Were the tools steam-cleaned?

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Were the tools rinsed with distilled water?

If the tools have visible oil or dirt that cannot be removed with conventional decontamination techniques were they sprayed with hexane?

Were the tools wrapped in foil for storage or transportation to prevent contamination?

6. SAMPLE COLLECTION PROCEDURES

Was the static water level measured to within 0.01-foot with an electronic water level probe prior to purging?

Were the static water levels measured in upgradient wells first?

Was a second confirmation reading taken?

Were all observations and data recorded on field sampling forms?

Was the electronic water level indicator rinsed with deionized water prior to and after each use?

Was all purge and sampling equipment decontaminated prior to use?

Were the monitoring wells purged with a stainless steel bailer, Teflon bailer, 1/2 hp submersible pump, 2-inch submersible pump or Well Wizard?

If a pump was used to purge and/or collect the sample, was the gas-powered generator at least 10 feet downwind during sampling?

Was a minimum of three casing volumes purged from the wells?

Were conductivity, temperature and pH monitored periodically during purging?

Were the well sampled after the conductivity, temperature and pH stabilized (within 10% variability between consecutive well casing volumes), and at least three well casing volumes were removed?

Was sampling equipment protected from ground contact by a plastic drop cloth and a decontaminated container?

Were the samples collected with either a stainless steel, Teflon bailer, 1/2 hp submersible pump or Well Wizard?

Was a new length of polypropylene rope used?

Were new latex gloves worn by field personnel during sampling of each well?

Were the monitoring wells sampled at a sufficiently slow rate to prevent agitation inside the well?

Were the contents of the sampling devices transferred to sample containers in a way that minimized agitation and aeration?

Detroit Coke RFI-RA QAPP

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Was the ground water at the purge wells sampled via a ball-valve tap at a low flow rate?

At the purge wells, was the sample port opened and ground water allowed to flow for three to five minutes prior to sample collection?

Were the samples for VOC analysis collected first?

Was the volume of sample collected for VOC analysis of sufficient volume to eliminate headspace in the sample container?

Was the rate of flow of water from the bailer into the sample container less than 100ml/minute?

Were ground water samples to be analyzed for dissolved metals filtered at the time of sampling, using a peristaltic pump with an in-line filter?

Were the metals samples preserved with dilute nitric acid?

Were additional ground water samples collected for field analysis of pH, conductivity, and temperature?

Were the probes for field analyses kept from contacting ground water samples for laboratory analyses?

Were the pH and conductivity equipment properly calibrated to the manufacturer's specifications?

Were the meters calibrated daily before use?

Were calibration checks performed after every 10 samples or less?

Were the calibration checks documented on the field forms and in the field log books?

Were all field measurements documented?

Was all purge water treated on-site using the existing ground water remediation system?

Were critical spare parts such as tape, pH probes and batteries kept on-site to reduce down time?

Were backup instruments and equipment available on-site or within a one-day shipment to avoid delays in the field schedule?

7. SOIL BORINGS

Were the soil borings drilled using a 4.25-inch inner diameter hollow-stem auger?

Was a 140-pound hammer free-falling 30 inches used to drive the 3-inch outside diameter, split spoon sampler 24-inches into the undisturbed soil ahead of the lead auger or open borehole?

Were the soil samples collected with a 2-foot long split-spoon sampler in accordance with ASTM Method D1586?

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Was the split spoon sampler decontaminated after each sample interval?

Did the field technician log the following information for each sampling interval:

Soil type? Soil depth? Soil consistency? Blow counts?

Was a portion of the sample placed into the inner of two zip-lock storage bags or into an air tight glass jar as soon as the split-spoon sampler was opened?

Was the sample allowed to sit for at least two minutes and allowed to reach room temperature in the sealed container?

Was the head space in the bags then analyzed with a FID/PID?

Was the FID/PID reading recorded on the boring logs?

Was the sample with the highest FID/PID reading and/or greatest visual evidence of impact submitted for laboratory analysis?

Was one sample from each shallow soil boring submitted for laboratory analysis?

Were two samples from each deep soil boring submitted for laboratory analysis?

For the deep borings, was one of the samples collected from the first interval with no indication of impact?

Were the soil samples from the deep borings visually inspected for DNAPLs?

Were the deep soil borings grouted using a cement bentonite grout (95% cement and 5% bentonite) tremied in place?

Appendix D

Earth Tech ELD, QA/QC Procedures Manual

Appendix D	Ap	pen	dix	D
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EARTH TECH ELD, QA/QC Procedures Manual

Quality Assurance Manual

Analytical Services

Prepared by: EARTH TECH 5555 Glenwood Hills Parkway Grand Rapids, MI 49588

July, 1994

EARTH TECH ANALYTICAL SERVICES QUALITY ASSURANCE/QUALITY CONTROL PROCEDURES MANUAL

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1.0 PURPOSE OF THE MANUAL

1.0 PURPOSE OF THE MANUAL

The purpose of this manual is to specify procedures and technical requirements to be used by EARTH TECH Analytical Services to assure that the data generated by the laboratory is accurate, reproducible and timely. This manual provides the chemistry laboratory a quality control plan which is to be used by every individual involved in the analytical efforts at EARTH TECH.

1.1 THE NEED FOR ANALYTICAL QUALITY CONTROL

There is a growing importance attached to the measurement of the concentration of any contaminant in water, effluents, and solid samples. As with any type of measurement the results of the methods utilized to measure the concentrations of these contaminants generally differ from the true concentration, i.e. all results are subject to error. Many experimental studies have shown that errors can arise which are as large as a 50 percent variations from the true value, and in fact, may vary between laboratories. Inaccurate analytical results restrict the ability of the analyst and the recipient of the data to draw valid conclusions and usually lead to false or misleading conclusions. Examples of common problems which arise during an analytical effort are as follows:

- A. Results, which are compared between two or more laboratories, are in error relative to each other.
- B. Results are to be used to decide if a water quality standard has been observed especially as the level of the analysis approaches the detection level.
- C. An inappropriate test procedure has been used to determine the analyte, resulting in values that do not represent the true sample concentration, i.e. direct aspiration of a turbid sample.

There is also increasing concern about the control of these errors being expressed at the local, the national and the international levels. The concern centers around the need to have a maximum amount of valid information obtained in a cost effective manner. In order to control errors, it is necessary to be able to measure the magnitude of these errors. This manual identifies the activities that are involved in the measurement and control of error. EARTH TECH considers analytical quality control of great importance, and requires that it be a primary feature in any analytical effort. EARTH TECH requirements for analytical quality control are in concert with the quality control needs and demands of other organizations, such as the Environmental Protection Agency, various state regulatory agencies, and private industry.

Approximately twenty to thirty percent of all the available effort for routine analysis is absorbed in the execution of quality control requirements. It is often argued that the extent of this effort is too great with respect to routine laboratories and their need to be

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profitable in their operation. The argument therefore claims that extensive quality control is an impractical expectation for a routine laboratory. However, the corporate policy at EARTH TECH demands that the appropriate level of quality control be applied to all analytical effort at EARTH TECH, regardless of the sample lot.

In the total effort, it is preferable to obtain twenty to thirty percent fewer results of known accuracy for each analytical batch than it is to obtain larger numbers of results of undefined accuracy. Due to the fact that all analytical procedures are subject to errors derived from many sources, it is not reasonable to assume that quality control is unnecessary with a "good" analyst. However, even a "good" analyst may not have an adequate idea of his (her) accuracy. Multiple studies by the EPA, both within laboratories and between laboratories, has shown this reasoning to be generally unsound.

2.0 QUALITY ASSURANCE ORGANIZATION AND RESPONSIBILITIES

2.0 QUALITY ASSURANCE ORGANIZATION AND RESPONSIBILITIES

2.1 OBJECTIVE OF THE QA PROGRAM AT THE EARTH TECH LABORATORY

The purpose of the quality control program is to continuously monitor error, both random and systematic, which inhibits the production of reliable and defensive analytical data. Error is inherent in any analytical routine, even with the most rigorous controls, and thus, a good QA program addresses not only the basic control techniques but also statistical means of measuring precision and accuracy and the confidence limits on these measurements.

The purpose of this manual is to specify the procedures, records, Chain-of-Command, and technical requirements which will be adhered to by the laboratories of EARTH TECH.

2.2 ORGANIZATION

Quality Assurance at the EARTH TECH Laboratory begins with the bench analyst and moves up through the Chain of Command ultimately residing at the level of the President. A QA program which is administered only at the upper levels of management is doomed to failure and is unfair to the bench level analyst who needs a means by which he can observe the quality of his work. Quality Assurance is a two way program at EARTH TECH where directives from management are as important as suggestions and assistance from the bench analyst.

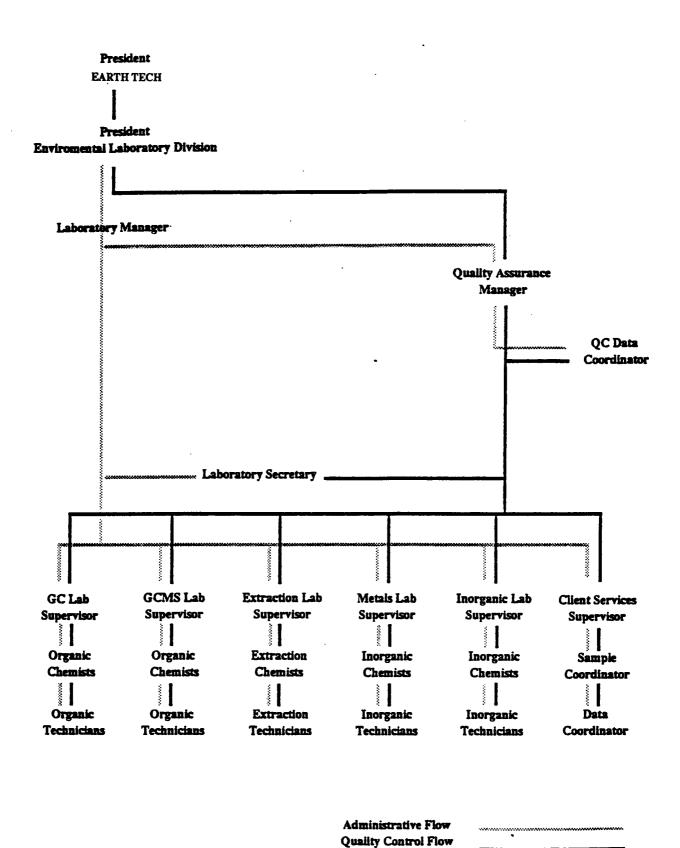
2.2.1 QA Chain of Command Flow Chart

The following flow chart represents both the QA Chain of Command (solid line) and the Administrative Chain of Command (dotted). The flow chart represents the philosophy of EARTH TECH relative to the interaction of QA and production. Although the QA Supervisor reports to the Director of Analytical Services in the supervisory Chain-of-Command, his responsibilities for quality control require that he answer to the President of the Environmental Laboratory Division. The QA Supervisor acts as an immediate record keeper, QA administrator and liaison to the lab manager.

However, when a question relative to the quality of analytical data arises, the QA Supervisor, in conjunction with the President of the Environmental Laboratory Division, has the right to prevent data dissemination. In cases of conflict, the President of the Environmental Laboratory Division has final authority except when a compromise or directive is issued by the President of WW Engineering & Science.

2.2.2 Responsibilities of the Laboratory Quality Assurance Supervisor

QUALITY CONTROL CHAIN OF COMMAND FLOW CHART



- 2.2.2.1 To monitor the Quality Assurance activities in the laboratory insuring adherence to all policies and procedures.
- 2.2.2.2 To identify problem areas and help in recommending improvement and changes.
- 2.2.2.3 To keep abreast of changing development in analytical Qc particularly requirements set by regulatory agencies.
- 2.2.2.4 To arrange or produce random blind control samples.
- 2.2.2.5 To approve all laboratory data prior to recording such data for report generation purposes.
- 2.2.2.6 To maintain QA/QC on all analytical activities and update control limits in a timely manner.
- 2.2.2.7 To oversee the maintenance of balance and controlled temperature apparatus record books on a daily basis and insure that such records are maintained on every piece of equipment.
- 2.2.2.8 To assure that bottle preparation, approval and storage meet established criteria.

2.2.3 Responsibilities of the Sample Coordinator

A full position description of the Sample Coordinator can be found in the "EARTH TECH Sample Receiving SOP".

- To insure that all samples received at EARTH TECH are properly preserved, split, logged-in, and stored in agreement with the log-in manual.
- To insure that all COC shipments are handled according to established procedures including storage, sample tracking and completion of files.
- To insure that all project sheets and subsequent paperwork is completed and filed.
- To insure that labile samples are distributed in a timely manner.

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• To cooperate with the QC Supervisor in introducing blind samples.

2.2.4 Responsibilities of the Analytical Staff

- To insure that all records are generated and recorded on a daily basis.
- To insure that the following bench level QC requirements are met. To fill out lab notebooks daily as required. To provide for QC on every batch of samples. This level of effort generally includes:

- 1) An initial calibration blanks and standards
- 2) 20% sample matrix spikes
- 3) 20% sample matrix duplicates
- 4) Laboratory control sample and a method preparation blank for each batch.
- To insure that instruments are calibrated prior to initiating any analysis and that no analyses are started unless calibration has been satisfactorily completed.
- To insure that every batch of analyses meets established QC guidelines or is reanalyzed automatically.
- To inform the lab manager of any reoccurring problems or systematic trends which may effect quality.

2.2.5 Responsibilities of the Laboratory Supervisor

- To insure that sufficient competent staff is available to administer QC.
- To insure that all participating analysts are certified in the test they are performing.
- To insure that effective training and orientation takes place for every new analyst.
- To insure that all QC procedures, directives or project oriented requirements are met.
- To review all preliminary reports and approve them prior to the generation of a final report.
- To interface with the QC coordinator and QA Supervisor on a routine and consistent basis.
- To take responsibility for immediate solutions to QA problems which may slow or stop production.

2.2.6 Responsibilities of the Data Coordinator

The Data Coordinator's (DC) responsibilities are:

- To enter all data generated into the appropriate records.
- To insure that the Laboratory Supervisor has signed the data forms (bench sheets prior to entry).
- To inform the QA Supervisor when a project is complete and ready for a preliminary report.
- To provide corrections to all reports from preliminary report feedback.

3.0 FACILITIES AND EQUIPMENT

3.0 FACILITIES AND EQUIPMENT

- 3.1 The physical plant layout diagram is enclosed. The approximate square footage allocated to each analysis area is presented as well as the number of personnel normally working in each area. A listing of equipment presently utilized by the EARTH TECH Laboratory is also enclosed.
- 3.2 The quality of the analytical instrumentation utilized by EARTH TECH Analytical Services is of great importance considering its ultimate effect on data quality. The following guidelines exist for the procurement of analytical instrumentation:

3.2.1 Equipment Need

An equipment need is identified by the Lab Manager or the President as a result of:

- o New Contractural Effort
- o Regulatory Changes
- o Normal Upgrade/Replacement
- o Capacity Improvements

3:2:2 Procurement Procedure

The performance specifications defined by the need are used to identify prospective equipment suppliers. The Lab Manager mails the performance specifications to the prospective equipment suppliers. Those suppliers able to meet the performance specifications are asked to provide a quotation for the purchase or lease of the equipment. An evaluation of the quotations is made by the Lab Manager with consideration given to such items as: equipment ease of use, degree of automation, specification compliance, potential for computerization, price and space requirements.

A written recommendation by the Lab Manager is presented to the President for their review and comment.

A final recommendation by the President of the Laboratory Division is made to the Vice President of Corporate Finance. The final approval is granted based on the assurance of complying with all regulatory and corporate guidelines for the generation of the highest quality data.

3.3 CHEMICAL PROCUREMENT AND INVENTORY PROCEDURE

All reagent specifications are dictated by the EPA/APHA or NIOSH approve analytical methods. These reagent specifications are identified and maintained on the chemical inventory index card system. The chemical inventory system assures the order of

chemical use and minimizes the possibility of exceeding their useful shelf life. The addition of a new method or a change in an existing method that requires a corresponding addition or change in a reagent used for that method will be identified by the Lab Area Supervisor or Group Leader. The Supervisor or Group Leader will update the chemical inventory.

All reagent specifications including available vendors are listed in the chemical inventory tables.

All chemical reagents are received by a representative from the appropriate lab area. The representative opens the shipping package and compares the packing slip with the contents. Discrepancies are identified to the Area Supervisor. The materials receipt is identified and recorded on the chemical inventory. The materials are then inventoried on the chemical inventory index. The index system identifies the amount(s) received and when. When the last bottle/container of the chemical remains, the chemical is placed on an open order sheet located in the lab.

The group leader/supervisor for that lab area is responsible for picking up the chemical open order sheet each week and preparing a purchase order. The purchase order is approved by the Lab Manager and a typed purchase order is issued to the approved vendor that has been previously identified as being able to supply the specified material. The receipt of the new order initiates the inventory system activities.

3.4 PREVENTATIVE MAINTENANCE

Every analytical instrument has a separate maintenance log book as identified in the Document Control Section No. 7.3.9. The required maintenance activities have been developed by the Lab Manager and each Group Leader/Area Supervisor. The maintenance activities comply with manufacturer specifications and working experience requirements.

Each maintenance log book contains a table indicating the frequency and type of maintenance required. The maintenance activity is documented each day or as the frequency requirements dictate.

Analysts are assigned the responsibility of maintaining various instruments or equipment in their respective laboratory areas. The Group Leader or Area Supervisor is responsible for checking the maintenance log books each week and signing off as checked. The Quality Assurance Supervisor is notified of any deviations or lack of maintenance activity performance, and corrective actions are taken.

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ANALYTICAL EQUIPMENT EARTH TECH-INORGANIC

			Year
Instrument Description	Manufacturer	Model No.	Purchased
Auto-Analyzer (dual channel)	Bran & Lubbe	TRAACS 800	1987
Auto-Analyzer (four channel)	Lachat	Quick Chem	1990
Conductivity Meter	YSI	32	1986
FTIR Spectrophotometer	Perkin-Elmer	1600	1990
pH/mv Meter	Beckman/Altrex	70	1989
pH/mw/ISE Meter	Orion	EA920	1991
Spectrophotometer (UV-VIS)	Shimadzu	1604	1990
Spectrophotomer (UV-VIS)	Shiamdzu	1201	1992
Total Organic Carbon Analyzer (TOC)	O.I.C.	700	1990
Total Organic Halogen Analyzer (TOX)	Xertex/Dohrman		1986
Turbidimeter	НАСН	43900	1992
Polarograph	EG&G Princeton	384B	1991
	Applied Research		
Auto-Titrator	Metler	DL12	1992

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ANALYTICAL EQUIPMENT EARTH TECH-METALS

			Year
Instrument Description	Manufacturer	Model No.	Purchased
AA Spectrophotometer (flame/furnace)	Perkin Elmer	5100 PC	1989
AA Spectrophotometer (furnace)	Perkin Elmer	5100 PC	1990
AA Spectrophotometer (furnace)	Perkin Elmer	4100 PC	1992
ICP Spectrophotometer	Perkin Elmer	Plasma 40	1989
ICP Spectrophotometer	Perkin Elmer	Optima 3000	1993
Autosampler	Perkin Elmer	AS-50	1989
Autosampler	Perkin Elmer	AS-51	1990
Autosampler	Perkin Elmer	AS-60	1992
Autosampler	Perkin Elmer	FIAS-200	1989
Mercury Amalgam System	Perkin Elmer		1990
Automated Mercury Analyzer	Leeman Labs	PS200	1992
Automated Mercury Preparation System	Leeman Labs	AP200	1992
Microwave Digestion System	CEM	MDS 810	1989

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ANALYTICAL EQUIPMENT EARTH TECH-ORGANIC

	•		Year
Instrument Description	Manufacturer	Model No.	Purchased
Gas Chromatographs w/ECD	Varian	3700	1986
Gas Chromatographs w/ECD	Varian	3700	1987
Gas Chromatographs w/ECD	Varian	3700	1988
Gas Chromatographs w/FID/ECD	Varian	3700	1988
Gas Chromatograph w/FID/CED	Varian	3400	1989
Gas Chromatograph w/Hall-PID	Tracor	540	1990
Gas Chromatograph w/Hall-PID	Tracor	585	1991
Gas Chromatograph w/Hall-PID	Tracor	9000	1992
Gas Chromatograph w/FID	HNU	301	1986
Autosampler	Varian	ALS 2016/LCS 2000	1988
Autosampler	Varian	8000	1991
Autosampler/Concentrator	Tekmar	ALS 2016/LCS 2000	1989
Autosampler/Concentrator	Tekmar	ALS 2016/LCS 2000	1989
Autosampler/Concentrator	Tekmar	ALS 2016/LCS 2000	1990
Autosampler	Tekmar	ALS 2050	1989
Gas Chromatograph	Perkin-Elmer		1993
Field GC's:			
Gas Chromatograph (FID/ECD)	Trimetrics	9000	1992
Gas Chromatograph (Hall/PID)	SRI	8610	1993
Gas Chromatograph (PID)	Photovac	10555	1991

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ANALYTICAL EQUIPMENT EARTH TECH-ORGANIC

			Year Date
Instrument Description	<u>Manufacturer</u>	Model No.	Purchased
Autosampler Concentrator	Tekmar	ALS 2016/LCS 2000	1989
Autosampler/Concentrator	Tekmar	ALS 2016/LCS 200	1989
Autosampler/Concentrator	Tekmar	ALS 2016/LCS 2000	1991
Thermal Tube Desorber	Envirochem, Inc.	850	1990
HPLC	Isco	2300	1988
HPLC	Perkin Elmer	Series 410	1992
LC Oven	Perkin Elmer	101	1992
Diode Array Detector	Perkin Elmer	235	1992
Fluorescence Detector	Perkin Elmer	LC240	1992
Chromatography Data Systems:			
Turbochrom	Perkin Elmer		1993

ANALYTICAL EQUIPMENT EARTH TECH-ORGANIC

	•		Year
Instrument Description	Manufacturer	Model No.	Purchased
Ion Trap	Varian	Saturn II	1991
Ion Trap	Varian	Saturn II	1991
Ion Trap	Varian	Saturn II	1993
Mass Spectrometer	Extrel	ELQ-400	1988
Mass Spectrometer	Extrel	ELQ-400	1989
Autosampler	Leap	CTC A2005	1993
Autosampler	Leap	CTC A2005	1993
Autosampler	Leap	CTC A2005	1993
Gas Chromatograph w/FID (screening)	Varian	3400	1991
Autosampler/Concentrator	Tekmar	ALS 2016/LCS 2000	1991
Autosampler	Dynatech	Dynasoils	1992
Autosampler	Dynatech	Dynasoils	1992
Autosampler	Dynatech	Dynasoils	1992
Chromatography Data Systems:			
Turbochrome	Perkin Elmer	3.3	1993

COMPUTER EQUIPMENT

EARTH TECH-ENVIRONMENTAL LABORATORY

Hardware

Hardware Description	Qty	Manufacturer	Model No.
Servers/Computers			
UNIX Workstation/Database Server	1	DEC	DEC System 5100
Novell File Server	. 1	DEC	486
Personal Computer	24	IBM Compatible	486
Personal Computer	28	IBM Compatible	386
Mini Computer	2	DEC	PDP-11/73
Printers			
Line Printer	1	Printronix	P6280L
Line Printer	2 .	Printronix	P6040
Laser Printer	1	HP	Laser Jet Series IV
Laser Printer	2	HP	Laser Jet IIISI
Laser Printer	1	HP	Laser Jet IIID
Laser Printer	4	HP	Laser Jet III
Laser Printer	1	HP	Laser Jet Series II
Dot Matrix Printer	10	Epson	FX-850
Scanning/Archiving Station			
Image Scanner	1	Fujitsu	M3903E
Optical Disk Drive	1	Matsushita	RF5010S
Page Monitor	1	Puretek	PT-15FPG
Computer	1	Everex	486SX/20
Networking Products			
802.5 Token Ring NIC	50	Thomas Conrad	TC4045
802.5 Token Ring MAU	3	Thomas Conrad	TC4050
Terminals	6	DEC	VT320
Terminal Servers	2	DEC	DEC Server 200

7/94

COMPUTER EQUIPMENT

EARTH TECH-ENVIRONMENTAL LABORATORY

Software

	•
<u>Developer</u>	<u>Version No.</u>
Oracle	6.0.36
EARTH TECH	1.4
DEC	ULTRIX 4.2
DEC	RSX11M V4.3
Novell	3.11
Novell	LAN Workplace for DOS 4.1
Microsoft	5.0
Microsoft	3.1
Oracle	6.0
Micro Focus COBOL	1.2.13
Microsoft	Word for Windows 2.0c
Microsoft	Excel 4.0
Lotus	123 3.1
Symantec	Q&A 4.0
Keaterm	Keaterm 420 2.0
Shapeware Corp.	Visio 2.0
Chemsoft	2.0
JT Baker	
Perkin Elmer	Turbo Chrome 3.3
PE Nelson	P400 V4.0
PE Nelson	P5100 V7.1
PE Nelson	P4100ZL V6.2
Varian	Saturn II Rev. C
Extrel	8.2
Quik Chem	Lachet Rel 22
Bran Lubbe	Traacs Ver. 3.02
•	
Courtland	1.01.02.07
Courtland	
CIS	
	Oracle EARTH TECH DEC DEC Novell Novell Microsoft Microsoft Microsoft Microsoft Lotus Symantec Keaterm Shapeware Corp. Chemsoft JT Baker Perkin Elmer PE Nelson PE Nelson PE Nelson Varian Extrel Quik Chem Bran Lubbe Courtland Courtland

PHYSICAL PLANT:

Laboratory Name: EARTH TECH Analytical Services

Address: 5555 Glenwood Hills Parkway SE

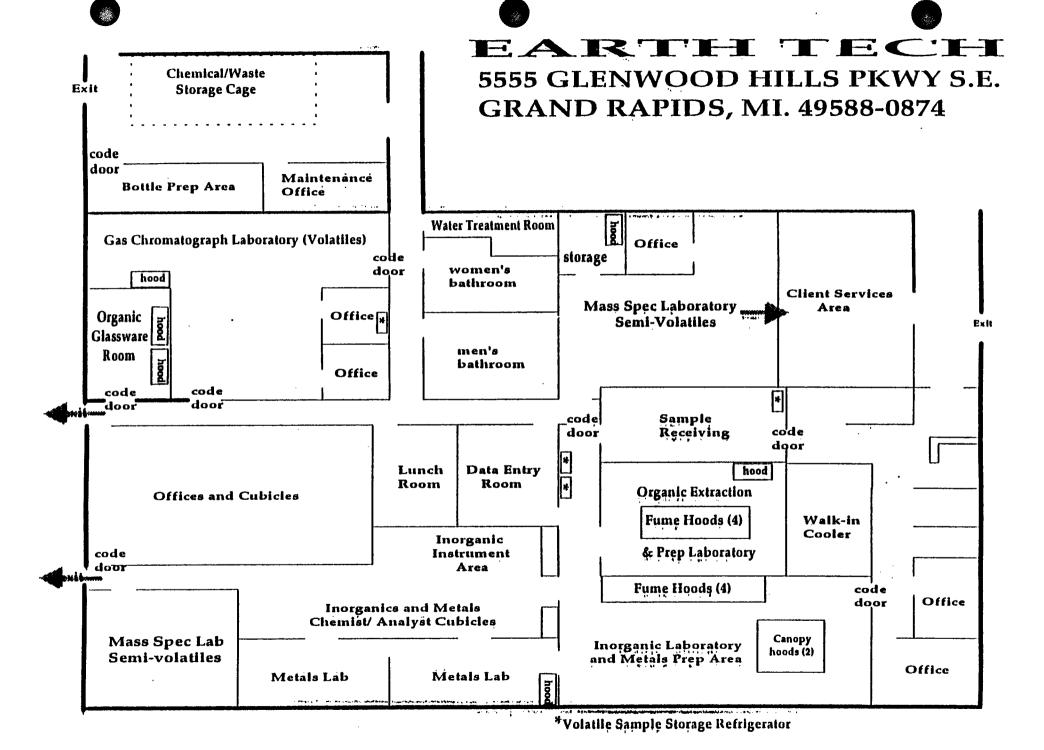
Grand Rapids, MI 49588

Name of Laboratory Manager: John P. Dullaghan

An attached drawing of the laboratory incidates the general areas of analysis, the space allotted to each, and the number of personnel generally assigned to each area.

Analysis	Space Allotted, Ft ²	No. of Personnel (August, 1994)
Wet Chemistry/Microbiology	Approx. 2000	11
Atomic Absorption/Emission	Approx. 2000	10
Volatile Organics	Approx. 2400	12
Semi-Volatile Organics	Approx. 1500	7
Sample Processing & Storage	Approx. 1500	4
Administrative Offices	Approx. 2500	26
Organic Pretreatment	Approx. 1000	6

74-Full Time 2-Part Time 76-Total



4.0 ANALYTICAL METHODOLOGIES

4.0 ANALYTICAL METHODOLOGIES

4.1 METHODS UTILIZED

EARTH TECH Laboratory maintains and updated reference volumes of approved analytical methodologies for environmental and non-environmental analysis. A responsibility of the Lab Manager and the President of the Environmental Laboratory is to continually seek and review regulatory method changes and their impact on current laboratory practices. The most commonly referenced materials include:

- "Methods for Chemical Analysis of Water and Wastes" EPA-600/4-79-020 revised March, 1983.
- "Manual of Analytical Procedures" NIOSH, Volumes 1 & 2, Third Edition, Feb., 1984.
- Standard Methods for the Evaluation of Water and Wastewater 17th Edition, APHA, AWWA, WPCF; 1989..
- "Handbook for Analytical Quality Assurance in Water and Wastewater Laboratories", EPA 600/4-79-019, March 1979.
- "Physical and Chemical Methods for the Evaluation of Solid Waste" EPA-SW846 Third Edition, 1990.
- "Guidelines Establishing Text Procedures for the Analysis of Pollutants". CFR July 1, 1990.

4.2 METHOD CALIBRATION AND OPERATING PROCEDURES

A standard operating procedure manual exists for all analytical procedures. The S.O.P's include specific calibration procedures that must be followed by an analyst prior to conducting sample analysis. The analyst is required to perform and document the calibration procedure. The calibration activity is identified by each analyst in their lab notebooks. The actual standards utilized are found in each instrument log book. It is the responsibility of each analyst to document all calibration and operating procedures utilized in the instrument log books. It is the responsibility of the group leaders and/or supervisors to notify the Quality Assurance Supervisor when deviations occur so that corrective actions can be taken. The corrective action will be to identify whether the information is simply missing (not entered) and to have it recorded or if the calibration has not been performed, to not release data generated that day and require those samples to be rerun.

5.0 METHOD CERTIFICATION

5.0 METHOD CERTIFICATION

All methods used by EARTH TECH Laboratories are certified prior to their use. Method Certification is contiguous with the certification of the analyst and requires essentially the same analytical program. Method certification is necessary in order to establish detection limits, method application limits and criteria for control limits. In most cases, detection limits and recoveries stated in a method are obtained under ideal conditions and do not reflect real world solutions, i.e., silty well water and industrial effluent versus a drinking water supply. Method certification falls into 2 categories: 1) Methods being employed for the first time and 2) Methods which are to replace currently certified methods (replacement methods). In either case, analysis of client sample may not proceed until certification has occurred.

5.1 METHOD CERTIFICATION

5.1.1 Linear Range

The first step in certifying a method is to establish the linear range (operating range) of the method. A method may be used only over the range in which it is linear. Some methods do not have linear ranges but curves from which results are calculated. For the moment we will ignore methods with curves. A linear range must be established independent of the method data since instruments can effect the range. Standards and multiple detections will be used for establishing the linear range. For example, a range of 1 to 1000 has 3 decades (3 orders of magnitude or 103). Therefore, a range of 1 to 1000 requires 11 levels of test standards (.5, 1, 2, 5, 10, 20, 50, 100, 200, 500, 1000). Notice that each decade follows the 0.5x to 10x rule, i.e. the area 10 to 100 is covered by 5,10,20,50 and 100. The range to be attempted is dependent on the method, the instrument and the analytical supervisor. If the responses show linearity, the range has been established. If, however, a curve develops or there appear to be two linear ranges, the standards must be repeated including additional levels to verify the status of the questionable area.

5.1.2 Working Curves

Some methods operate from a curve response, i.e. sodium by emission spectroscopy. The method will indicate the working curve which must be verified. The method with working curves requires a full curve each time an analysis is to be performed.

5.1.3 The Generation of the Method Detection Limit

The method detection limit (MDL) is defined as the minimum concentration of a substance that can be identified, measured and reported with 99% confidence that the analyte concentration is greater than zero and determined from analysis of a

sample in a given matrix containing analyte.

SCOPE AND APPLICATION

This procedure is designed for applicability to a wide variety of sample types ranging from reagent (blank) water containing analyte to wastewater containing analyte. The MDL for an analytical procedure may vary as a function of sample type. The procedure requires a complete, specific and well defined analytical method. It is essential that all sample processing steps of the analytical method be included in the determination of the method detection limit.

The MDL obtained by this procedure is used to judge the significance of single measurement of a future sample.

The MDL procedure was designed for applicability to a broad variety of physical and chemical methods. To accomplish this, the procedure was made device or instrument independent.

PROCEDURE

- 1. Make an estimate of the detection limit using one of the following:
 - (a) The concentration value that corresponds to an instrument signal/noise ratio in the range of 2.5 to 5. if the criteria for qualitative identification of the analyte is based upon pattern recognition techniques, the least abundant signal necessary to achieve identification must be considered in making the estimate (PCB).
 - (b) The concentration value that corresponds to three times the standard deviation of replicate instrumental measurements for the analyte in reagent water.
 - (c) The concentration value that corresponds to the region of the standard curve where there is a significant change in sensitivity at low analyte concentrations i.e. a break in the slope of the standard curve.
 - (d) The concentration value that corresponds to known instrumental limitations.

It is recognized that the experience of the analyst is important to this process. However, the analyst must include the above considerations in the estimate of the detection limit.

- 2. Prepare reagent (blank) water that is as free of analyte as possible. Reagent or interference free water is defined as a water sample in which analyte and interferent concentrations are not detected at the method detection limit of each analyte of interest. Interferences are defined as systematic errors in the measured analytical signal of an established procedure caused by the presence of interfering species (interferent). The interferent concentration is presupposed to be normally distributed in representative samples of a given matrix.
- 3. (a) If the MDL is to be determined in reagent water (blank) prepare a laboratory standard (analyte in reagent water) at a concentration which is at least equal to or in the same concentration range as the estimated MDL (Recommend between 1 and 5 times the estimated MDL) Proceed to Step 4.
 - (b) If the MDL is to be determined in another sample matrix, analyze the sample. If the measured level of the analyte is in the recommended range of one to five times the estimated MDL proceed to Step 4.

If the measured level of analyte is greater than five times the estimated MDL, add a known amount of analyte to bring the concentration of analyte to between one and five times the MDL in the case where an interference is co-analyzed with the analyte.

If the measured level of analyte is greater than five times the estimated MDL there are two options:

- (1) Obtain another sample of lower level of analyte in same matrix if possible.
- (2) The sample may be used as is for determining the MDL if the analyte level does not exceed 20 times the MDL of the analyte in reagent water. The variance of the analytical method changes as in the analyte concentration increases from the MDL, hence the MDL determined under these circumstances may not truly reflect method variance at lower analyte concentrations.
- 4. (a) Take a minimum of seven aliquots of the sample to be used to calculate the MDL and process each through the entire analytical method. Make all computations according to the defined method with final results in the method reporting units. If blank measurements are required to calculate the measured level of

analyte, obtain separate blank measurements for each sample aliquot analyzed. The average blank measurement is subtracted from the respective sample measurements.

- (b) It may be economically and technically desirable to evaluate the estimated MDL before proceeding with 4a. This will: (1) prevent repeating this entire procedure when the costs of analyses are high and (2) insure that the procedure is being conducted at the correct concentration. It is quite possible that an incorrect MDL can be calculated from data obtained at many times the real MDL even though the background concentration of analyte is less than five times the calculated MDL. To insure that the estimate of the MDL is a good estimate, it is necessary to determine that a lower concentration of analyte will not result in a significantly lower MDL. Take two aliquots of the sample to be used to calculate the MDL and process each through the entire method, including blank measurements as described above in 4a. Evaluate these data:
 - (1) If these measurements indicate the sample is in the desirable range for determining the MDL, take five additional aliquots and proceed. Use all seven measurements to calculate the MDL.
 - (2) If these measurements indicate the sample is not in the correct range, re-estimate the MDL, obtain new sample as in 3 and repeat either 4a or 4b.
- 5. Calculate the variance (S²) and standard deviation (S) of the replicate measurements, as follows:

where the x1i = 1 to n are the analytical results in the final method reporting units obtained from the n sample aliquots and X,2 refers to the sum of the X values from i = 1 to n.

6. (a) Compute the MDL as follows:

$$MDL = t_{(n-1),-a} = .99$$
). S

where:

MDL - the method detection

t(n-1,1-a = .99) = the students' t value appropriate for a 99% confidence level and a standard deviation estimate with n-1 degrees of freedom. See Table.

S = standard deviation of the replicate analyses.

(b) The 95% confidence limits for the MDL derived in 6a are computed according to the following equations derived from percentiles of the chi square over degrees of freedom distribution (X2/df) and calculated as follows:

MDLucl = 0.64 MDL MDLucl = 2.20 MDL

where MDLlcl and MDLucl are the lower and upper 95% confidence limits respectively based on seven aliquots.

- 7. Optional iterative procedure to verify the reasonableness of the estimated MDL and calculated MDL of subsequent MDL determinations.
 - (a) If this is the initial attempt to compute MDL based on the estimated MDL in Step 1, take the MDL as calculated in Step 6, spike in the matrix at the calculated MDL and proceed through the procedure starting with Step 4.
 - (b) If the current MDL determination is an iteration of the MDL procedure for which the spiking level does not permit qualitative identification, report the MDL as that concentration between the current spike level and the previous spike level which allows qualitative identification.
 - (c) If the current MDL determination is an iteration of the MDL procedure and the spiking level allows qualitative identification, use S2 from the current MDL calculation and S2 from the previous MDL calculation to compute the F ratio.

if $S^2_A/S^2_B < 3.05$

then compute the pooled standard deviation by the following equation:

Spooled =

if $S^2A/S^2B > 3.05$, respike at the last calculated MDL and process the samples through the procedure starting with step 4.

(c) Use the Spooled as calculated in 7b to compute the final MDL according to the following equation:

MDL = 2.681 (Spooled)

where 2.681 is equal to t(12,1-a=.99)

(d) The 95% confidence limits for MDL derived in 7c are computed according to the following equations derived from percentiles of the chi squared over degrees of freedom distribution.

MDLucl = 0.72 MDL MDLucl = 1.65 MDL

where LCL and UCL are the lower and upper 95% confidence limits respectively based on 14 aliquots.

REPORTING

The analytical method used must be specifically identified by number or title and the MDL for each analyte expressed in the appropriate method reporting units, if the analytical method permits options which affect the method detection limit these conditions must be specified with the MDL value. The sample matrix used to determine the MDL must also be identified with the MDL value. Report the mean analyte level with the MDL if a laboratory standard or a sample that contained a known amount analyte was used for this determination, report the mean recovery, and indicate if the MDL determination was iterated.

If the level of the analyte in the sample matrix exceeds 10 times the MDL of the analyte in reagent water, do not report a value for the MDL

REFERENCE

40 CFR Part 136 Appendix B, USEPA Chapter 1, 7/1/90.

Table of Students t Values at the 99 Percent Confidence Level

Number of Replicates	Degrees of Freedom (n-1)	t(n-1,1-oc=.99)
7	6	3.143
8	7	2.998
9	8	2.896
10	9	2.821
11	10	2.764
16	15	2.602
21	20	2.528
26	25	2.485
31	30	2.457
61	60	2.390
		2.326

5.1.4 Method Spikes

Method spikes will be carried out over 2 separate days at the specified levels including blanks and calibration standards. The data obtained at the 2x or 5x level (for each certified range) will be used to establish a mean and standard deviation for initial control charts. Once this data has been generated and approved by the analytical manager, the method has preliminary certification and is ready for application to real world samples. These control limits will be updated with every batch of samples until 30 numbers have been developed to establish reliable control limits. After 30 data points the DC will provide updated control limits with each additional 20 numbers.

5.2 REPLACEMENT METHOD CERTIFICATION

When a new method is to be employed (where a new method is defined as including a new instrument method, i.e. flame vs flameless AA or Hall vs ECD), the method must be certified prior to its use on client samples.

Certification follows the procedures described in Sections 5.1. The results of these tests are important but are not necessarily compared to the current method. The detection limit may change and the work range may change but if they meet the needs of the lab, these changes are to be ignored. One may elect to utilize a t-test analysis to identify the

method differences as being significant or not.

5.2.1 Comparison by the t-Test

One sample will be analyzed a minimum of 4 times by each method. The results will be subject to a t-test analyses. If the t-test indicates statistical correlation, regardless of the correlation coefficient, the new method is certified. If the t-test fails, refer to Section 5.2.2 below.

5.2.2 Decisions on Certification

The purpose of a new method is to improve accuracy, precision and efficiency. Efficiency is of no consequence if a method is imprecise and inaccurate, and therefore, is not a consideration in certifying new methods. However, a new method may fail the t-test because it is more accurate and/or more precise. Careful consideration and more analyses may be necessary with a new method by the analytical manager and supervisor.

6.0 ANALYST TRAINING AND CERTIFICATION

6.0 ANALYST TRAINING AND CERTIFICATION

6.1 RATIONAL

Consistent with requirements by the EPA and other regulatory agencies for analyst training and certification programs, EARTH TECH has a strict policy relative to the training and certification of analysts prior to their involvement in the analysis of client samples. The program is necessary in order to maintain continuity in all analytical programs and to insure the integrity of all data.

6.2 TRAINING

The supervisor is responsible for training all new personnel. This training will be in conjunction with the group (workstation) and group leader if applicable. Training will include, but not be limited to, EARTH TECH Analytical Services QA requirements, paperwork flow, lab safety and organizational structure. In addition, the new analyst will be given copies of the QA manual, log-in manual and methodologies which the analyst will be required to read. Training in the methods to be used will be initiated prior to analyst certification.

6.3 CERTIFICATION

Each new EARTH TECH analyst will be required to receive certification on all methods which he is to perform. Certification insures that the analyst can meet method detection limits and quality control limits as established for the method. Certification includes two parts, both of which must be completed satisfactorily.

6.3.1 Method Spikes

Analysis of spiked lab pure water at the levels of 0.5x, 1.0x, 2.0x, 5.0x and 10x where x is the established detection limit. This will include 2 blanks and a duplicated spike at 2.0x or 5.0x and will occur on 2 separate days. The data, where the duplicated results are averaged. These results must match current EARTH TECH Schwart control chart limits. Additional parameters such as consistent instrument calibration curves will be evaluated.

6.3.2 Check Sample Analysis

The analyst will test a known blind check sample in duplicate including a blank. All the data must fall within established control limits for the parameters.

6.3.3 Current Analysts Training

The LDI analyst, who is assigned a new method, must complete the certification program for the methods as outlined above prior to performing analyses on client

samples.

6.4 RECERTIFICATION

All EARTH TECH analysts will recertify on all their respective methods when required or demonstrated by two method spike performance failures following the procedures set forth in Section 6.3.1. The results must meet previous data, assuming that the same methods are employed.

6.5 PERFORMANCE AUDITS

The Laboratory Manager, in cooperation with the QA Supervisor, will perform individual audits on all aspects of the operation biannually. These audits will include recertification data, control limits, all levels of records and laboratory performance on all check samples and instituted blind QA samples. A report of the audit results including recommendations will be forwarded to the President of the Environmental Laboratory Division.

7.0 DOCUMENT CONTROL, FLOW AND STORAGE

7.0 DOCUMENT CONTROL, FLOW AND STORAGE

7.1 PURPOSE

The paperwork trail must be designed to insure that after the issuance of a report, anyone - a client, a lawyer or the President of EARTH TECH can track a single sample result back through EARTH TECH records to the origin of the standards used in calibration and the identity of the person who prepared the sample bottles.

7.2 PAPERWORK FLOW

As shown in the attached, "Flow Diagram" the paperwork trail is eventually the same for routine work as it is for samples under Chain-of-Custody. The general axiom is that a COC procedure is doomed to failure without a pre-existing scheme of tight sample and analytical control available as a routine measure. This contention, however, is only of minimal consequence with respect to the need for detailed records. The records trail can provide the following:

- Answers to questions of analytical integrity for results which are 2 months or two years old.
- · Assistance in finding and solving random and systematic problems.
- Assistance in preventing long term degradation of analytical integrity.
- Assistance in insuring continuity of analytical effort despite personnel and mechanical changes.

7.3 DOCUMENT REQUIREMENTS

The following subsection identifies all documents which are generated during the course of any project:

7.3.1 Project Sheets

Every sample or group of samples which enter the EARTH TECH facility must be accompanied by the appropriate project sheet which has been properly filled out and provided to the Sample Coordinator (SC). The SC may not log-in samples for which there are not project sheets or for which there the project sheet is incomplete. An example project sheet is attached as Figure 2.

7.3.2 New Project Approval Form

Projects which require testing or analyses not routinely provided at EARTH TECH must have prior approval on a Project Questionnaire and commitment from the Analytical Manager and the head of the appropriate analytical group(s). For the project manager's purpose, the approval forms insure that the analytical

testing area has received notification and will be prepared. For the analytical managers purpose, proper notification has been received and sufficient time has been allotted for preparation and development. Projects requiring rush turn around on modified methods must be approved as well. An example of a Project Ouestionnaire is attached as Figure 3.

7.3.3 Problem Project Sheets

When the Sample Coordinator (SC) identifies a problem with a sample shipment or project sheet, a Problem Project Sheet will be initialed and sent to the project manager for resolution. See Figure 4.

7.3.4 Chain-of-Custody Forms

There are three forms for Chain-of-Custody samples. All three forms must be properly completed and included in the project file for each and every COC project.

7.3.4.1 COC SHIPPING RECORD

The shipping record must be received in the shipping container with every COC shipment. The form attached as Figure 5 is similar to the form used by the EPA. This form will be used by EARTH TECH field samplers and returned with the samples. Other forms of a similar nature may be used by other clients. However, the information required on the EARTH TECH form must be present on any other client form or they run the risk of their COC being rejected as a continuous trackable COC event.

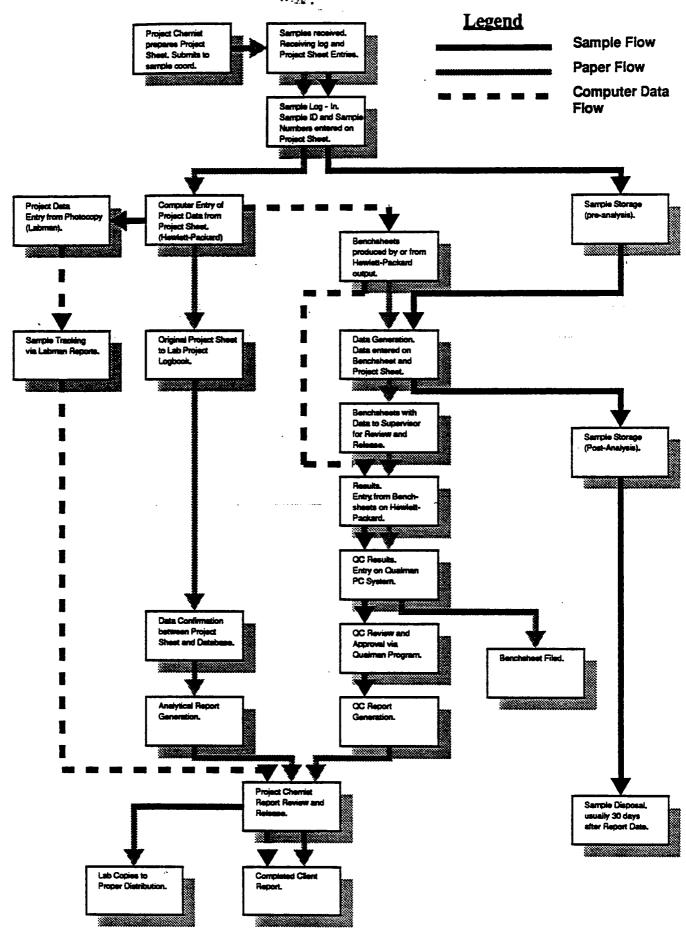
7.3.4.2 COC SAMPLE CONTROL RECORD

This form is used as a record of the movement of COC samples in and out of the COC locked storage. The analyst signs samples in and out each time a sample(s) is removed for any analysis. A copy of the form is attached as Figure 6. After all analyses are complete, the Sample Coordinator files the form in the COC project file.

7.3.5 Work Sheets/Project Sheets

Work sheets are the analytical assignment forms generated by the computer or the lab manager within 24 hours after log-in for each project or group of projects. The work sheets are divided into work stations, i.e. the analytes for which one or more analysts has sole responsibility. In many cases, the work sheets will have an entry position for the results of each analyses for each sample. In either case, the

Sample and Document Flow Diagram



ENVIRONMENTAL LABORATORY DIVISION Project Initiation

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Client Name	(32 characters available)	1 1 1 1 1	
Report Address	· <u> </u>	1111	
•		<u> </u>	
		1 1 1 1	
			<u> </u>
Billing Address		1 1 1 1 1 1	
(if different)			1 1 ! ! !
		1 1	
Client Alias		1111	
		•	
Client Contact	(20 characters available)	Spa	ace for the
Phone	([]	Clie	ent Narrative provided on
roject Chemist		the	back side of
Client Expiration (Date - Narrative		
Project Descriptio	n		
	(2 lines of 26 characters available)		
Price Code		☐ Price Fact	or L
Price Code Expire	re Date		
Project Contact			
Phone	(<u> </u>		
Project Expiration	n Date		
Purchase Order N	No Proj. T	ype ⁽¹⁾	Report Format ⁽²⁾
Contract No.	Field E	3lanks 📖	Methods Page
CS Mgr.	Case I	Narr. 📖	QC Report
CCS Project No.			

⁽¹⁾ C = Competitive Quote; D = Direct Request; R = Renewal (2) S = Standard; C = Cas# Report

Fre	quency (1)		Turnaround		Flame		Bottles (4)	
Sul	bmit/Yr.		C.O.C. (2)		Reactive		Carrier	
3	f Samples		QC Type (3)		Contact		Sample Storage	
					Health	L	Bottle Address (5)	لـا
	(2) I = Inter (3) RAS, Sa (4) H = Hol	d; S = Ship if Bottle Address is		Client Address		•	Narrative	
		bottle 5	inphing Addres	3			Space for the Project Narra is provided o the back side this sheet	ntive:
		escription		(26 characters		<u> </u>		
	Date Expec	ted LLL-L	<u> </u>					
*	Bottle Due	Date LLL - L						
	Turnaround	Days						
	Narrative							
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	Test Group Description	(40 characters available)	
	Sample Matrix		
	Date Expected	Bottle Due Date	
	Parameters	Method Number (Reference Citation)	Specific DL
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ENVIRONMENTAL LABORATORY DIVISION PROJECT QUESTIONNAIRE

REQUEST FOR WORK/QUOTATION (circle one)

Client	Proj. No)		
ClientProject Name		Proj. Mgr	Initials	:
(How do you want it	to look on the rep	oort)?	•	
Where should report Date of request of w	be routed?			
Date of request of w	ork?	Lab Notified	YES NO	
Date samples will ar Project Frequency:	rive in lab:			
Project Frequency:	One Time	Other (specify)	
Turnaround required	E	oue Date:	_Time:	
Confirmed in ELD b)y:	<u> </u>		
Job Description:				
Quality control requi				
Does QC need to be			_	
Is strict Laboratory			0	
Have sample contain				
Sample containers for		requested from		
Grand Rapids/Livon				
No. of water sample	es:			
Parameters required				
				
			······································	
Specific methods, de	etection limits and	i/or program require	ments (e a NPDES	
Act 307)	Joodon Mino, an	nor broßimit rodano		•
1100 307 ,				
				
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No. of soil samples:		·		
Parameters required		d (circle one):		
		· · · · · · · · · · · · · · · · · · ·		
Specific methods, de	etection limits, an	d/or program require	ments (e.g. RCRA,	Act
307, etc.)	,	•	-	
	····			
·			·	
				
NC-:				
No. of air samples:	-			

•	
•	
	Specific methods, detection limits, and/or program requirements (e.g. ACGITLV, etc.)
•	No. of other samples: Type: Parameters required are or/see attached (circle one):
٠	Parameters required are or/see attached (circle one):
•	
•	
٠	
٠	
	Specific methods, detection limits, and/or program requirements (e.g. Act 30 RCRA etc.)
	RCRA, etc.)
	• • • • • • • • • • • • • • • • • • • •
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	RCRA, etc.)
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	Hazard levels associated with the samples are: Has the client has been advised that any hazardous samples will be returned them? YES NO
	Hazard levels associated with the samples are: Has the client has been advised that any hazardous samples will be returned them? YES NO Disposal of samples will take place 21 to 30 days after report mailing unless noted otherwise (If otherwise is noted a charge of \$5/sa
	Hazard levels associated with the samples are: Has the client has been advised that any hazardous samples will be returned them? YES NO Disposal of samples will take place 21 to 30 days after report mailing unless noted otherwise (If otherwise is noted a charge of \$5/sa month will apply).
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	Hazard levels associated with the samples are: Has the client has been advised that any hazardous samples will be returned them? YES NO Disposal of samples will take place 21 to 30 days after report mailing unless noted otherwise (If otherwise is noted a charge of \$5/sa month will apply). Costs for the analysis were confirmed by (ELD) of the Gr. Rapids Branch. Is there any particular format needed for the final report? YES NO (If yes.)
	Hazard levels associated with the samples are: Has the client has been advised that any hazardous samples will be returned them? YES NO Disposal of samples will take place 21 to 30 days after report mailing unless noted otherwise. (If otherwise is noted a charge of \$5/sa month will apply). Costs for the analysis were confirmed by (ELD) of the Gr. Rapids Branch. Is there any particular format needed for the final report? YES NO (If yed discuss with ELD Project Chemist)
	Hazard levels associated with the samples are: Has the client has been advised that any hazardous samples will be returned them? YES NO Disposal of samples will take place 21 to 30 days after report mailing unless noted otherwise. (If otherwise is noted a charge of \$5/sa month will apply). Costs for the analysis were confirmed by (ELD) of the Gr. Rapids Branch. Is there any particular format needed for the final report? YES NO (If yet discuss with ELD Project Chemist) Are there any field measurements to be reported? YES NO
	Hazard levels associated with the samples are: Has the client has been advised that any hazardous samples will be returned them? YES NO Disposal of samples will take place 21 to 30 days after report mailing unless noted otherwise. (If otherwise is noted a charge of \$5/sa month will apply). Costs for the analysis were confirmed by (ELD) of the Gr. Rapids Branch. Is there any particular format needed for the final report? YES NO (If yet discuss with ELD Project Chemist) Are there any field measurements to be reported? YES NO If so specify Are you running field blanks? YES NO
•	Hazard levels associated with the samples are: Has the client has been advised that any hazardous samples will be returned them? YES NO Disposal of samples will take place 21 to 30 days after report mailing unless noted otherwise (If otherwise is noted a charge of \$5/sa month will apply). Costs for the analysis were confirmed by (ELD) of the Gr Rapids Branch. Is there any particular format needed for the final report? YES NO (If yet discuss with ELD Project Chemist) Are there any field measurements to be reported? YES NO If so specify
•	Hazard levels associated with the samples are: Has the client has been advised that any hazardous samples will be returned them? YES NO Disposal of samples will take place 21 to 30 days after report mailing unless noted otherwise

FIGURE 4

EARTH TECH LABORATORY PROBLEM PROJECT REPORT

SAMPLES RE	CEIVED ON AT AM/PM FROM:
AND DESCRI	BED AS WERE RECEIVED HAVING THE FOLLOWING
	EARTH TECH PROJECT APPROVAL FORM - ABSENT/INCOMPLETE
	CHAIN-OF-CUSTODY - ABSENT/INCOMPLETE
	CHAIN-OF-CUSTODY - DOES NOT MATCH SAMPLE TAGS
	SAMPLE BOTTLES - BROKEN
_	SAMPLES ABSENT - QUAN. DOES NOT MATCH APPROVAL FORM
	SAMPLE BOTTLES - INCORRECT FOR ANALYSIS
_	SAMPLE PRESERVATIVES - INCORRECT FOR ANALYSIS
	SAMPLE VOLUMES - INCORRECT FOR ANALYSIS
	SAMPLE TAGS - WRONG I.D./ABSENT
	FIELD FORMS - ABSENT/INCOMPLETE
-	CUSTODY SEALS - ABSENT/NOT INTACT
	NON-ROUTINE PROJECT - NO PRIOR APPROVAL
	S IN QUESTION WILL BE PROCESSED AS IS PLACED ON HOLD ORRECTIVE ACTIONS OR DIRECTIVES ARE ISSUED.
THANK YOU EARTH TECH SAMPLE COO	LABORATORY

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Project No. Project Name Samplers (signature)													9	Container Type & Volume		Method			
Samplers	(algna)	mel											No. of Containers	ritain Volum		t pod		Analysis Required/Comm	ent s
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	·			L		[<u> </u>	

^{*} MATRIX: WATER (WTR), WASTEWATER (WW), SOIL (SOL), SLUDGE (SLU), AIR, OIL, HAZARDOUS WASTE (HW)

work sheet, upon completion of all analyses, will be turned into the appropriate supervisor with the proper bench sheets attached. Unless specifically advised, data will not be accepted on any form other than the project approval form sheets.

7.3.6 Bench Sheets

The analysis of every analyte or group of analytes needed, i.e. VOA's requires a specific bench sheet which includes all results from the analysis of a group of samples. There are specific bench sheets for each analyte including specific requirements for their use. Examples of each bench sheet, can be found in Figures 7, 8 and 9.

7.3.7 Lab Notebooks

The lab notebooks are the daily records of all activities of an analyst, or group of analysts, working in the lab. The notebooks will be bound and paginated. The notebook will be cleanly labeled on the inside cover with the date issued, the analyst's name, and the date completed. There are several specific rules which will be follows:

- All entries are in ink
- There are no erasures, obliterations, or white outs allowed
- Corrections are single lined and initialed
- A new page is started each day or with every batch of samples
- Empty space is covered with a Z and signed and dated across the obtuse line
- · Any and all work, observations and errors are noted
- Problem areas identified

When the instrument has just been repaired, a lamp changed, new column installed, detector repaired, or changed in any other manner, the log will also contain:

- · A comment relative to the change or repair
- Reference page number to the Instrument Maintenance Log

The organic log books will also contain the following information relative to GC and GCMS oven and column conditions UNLESS they are exactly as specified in the referenced method which then will be commented on as such:

- · column used (packing, diameter, length, type) o capillary as split or splitless
- · current type and flow
- make up flow if appropriate
- oven temperature and program if appropriate

CCV: Stk

05-SEP-91

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METALS	BENCHSHEET

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Test #: 19 Parameter: COF Method: FLA Ref. Cit.: USE Comments:	ME/CU/UTR	Ú	ODL: 0.01 nit: mg/l							Benchs C Revi	Owner: late run: lewed By:	2462 1.6	and species pad for	
Client Submittal	Sample COC	. øç.	Reported Conc.	Duplicate Result	Spike Result	Spike Qty.	Spike Stock #	% dif	% rec	ODL.	Analyst	EXC	: DNR	
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Result

05-SEP-91 FRACTION Test #: 376.01- 35.01 Parameter: VOLATILE'S GC/MS 8240 Benchsheet ID: 2466 Method: VOL/P&T/MS/UTR Owner: Ref. cit.: USEPA-8240 Unit: Ug/l Date run: Client: 3M Company/St. Paul, Minnesota Project: 428 3M Request # J2496 Hain Plant Project: Instrument #: Volatile Organics Analysis Submittal: Est anal hrs: Sample: 1422 J2496-1 Act hrs: Expiration date: 05-8EP-1991 QC: RAS COC: Stock std #: Lab due date: 05-SEP-1991 Client due date: 12-SEP-1991 C=0 F=0 H=1 R=0 Supervisor: Parameter Result Parameter Result 1. ACETONE C 50 21. TRANS-1, 3-DICHLOROPROPENE C 4.0 2. BENZENE C 1.0 22. ETHYL BENZENE **C 1.0** BROMODICHLOROMETHANE C 2.0 23. 2-HEXANONE C 50 BROMOFORM ₹ 15 24. 4-HETHYL-2-PENTANONE C 50 BROMOMETHANE (10 25. HETHYLENE CHLORIDE (5.0 2-BUTANONE C 50 26. STYRENE C 10 7. CARBON DISULFIDE C 5.0 27. 1,1,2,2-TETRACHLOROETHANE C 2.0 8. CARBON TETRACHLORIDE C 4.0 28. TETRACHLOROETHENE C 2.0 9. CHLOROBENZENE C 1.0 29. 1, 1, 1-TRICHLOROETHANE C 2.0 10. CHLOROETHANE C 10 30. 1, 1, 2-TRICHLORDETHANE C 3.0 11. 2-CHLOROETHYLVINYL ETHER C 10 31. TRICHLOROETHENE C 2.0 12. CHLOROFORM C 1.0 32. TOLUENE € 1.0 13. CHLOROMETHANE C 10 33. VINYL ACETATE C 5.0 14. DIBROMOCHLOROMETHANE (3.0 34. VINYL CHLORIDE C 10 35. XYLENE(8) 15. 1,1-DICHLOROETHANE (2.0 C 5.0 16. 1,2-DICHLOROETHANE (2.0 17. 1,1-DICHLOROETHYLENE C 2.0 18. 1,2-DICHLOROETHENE(TOTAL) C 4.0

19. 1, 2-DICHLOROPROPANE

20. CIS-1, 3-DICHLOROPROPENE

C 3.0

C 4.0

*** SEMI-VOLATILES ORGANICS ***
Initial wt./vol.
Final volume
Dilution factor
**** VOLATILES ORGANICS ****
Initial wt./vol.
Volume purged
Dilution factor

Parameter

commonly required in dealing with analytical errors. There are a large number of text books dealing with statistics and this particular section does not attempt to replace these books. The intention is merely to present the essential aspects in the simplest manner possible. Certain approximations have been used when considered appropriate and no previous knowledge of statistics has been assumed. Should the analyst be interested in consulting additional texts for a more rigorous and detailed treatment of the subject, he is referred to the references at the end of section 9.0.

Analysts who are unfamiliar with statistical approach, may find this section on first glance rather complicated. In order to understand statistics for the QA function, it is important not to be put off by the first impression.

The fundamental statistical concepts are essentially simple and equivalent to the intuitive common sense, or perhaps scientific approach, adopted by any good analyst.

9.4.1 Random Error Distribution

If the results from the analysis of numerous aliquots of a homogeneous sample are plotted on a histogram, it is generally found that the proportion of the results deviating from the mean increased, i.e., as the deviation of the results from the mean grows broader. In other words, the probability of obtaining a random error of a given size decreases as the size of the error increases. The basis of statistical techniques is to quantitatively estimate the probabilities of errors of different sizes so that one can deduce the probable random error of a particular analytical result. If the analyst were to increase the number of analysis of a single sample indefinitely, and the size of the intervals used for plotting the histogram were decreased, the latter would tend to smooth the curve. This limiting curve is the frequency distribution of results and defines a relationship between the magnitude of the result and the probability of obtaining such a value. Throughout this manual, it will be assumed that the analytical results follow the normal distribution which is defined by the following equation:

$$p(x) =$$

Where:

- = the mean of all the conceptionally infinite number of results.
- = the standard deviation of results
- p(x) = the probability density which is interpreted by noting that the probability of obtaining a result between the values a & b is the area of the curve between those values.

and this interval can be evaluated given the equation for P(X).

The peak of this distribution curve occurs at x=u, the theoretically perfect mean established by an infinite number of results. The width (which is indicated by the scatter results) is determined solely by the standard deviation of the test. For example, 95% of the area under the curve, i.e. 95% of all results, is enclosed within the limits plus or minus 1.96. Such properties allow limits for the uncertainty of an individual analytical result to be calculated. Taking the current discussion, for example, on no more than 5 occasions in one hundred will the result differ from the mean u be more than 1.96. Thus, an analyst may attach to a result limits that define the range in which the true mean is expected to lie. The statement, R-1.96 is less than u which is less the R+1.96, is an accurate statement on 95% of all occasions. "R" in this particular case would stand for the result. By referring to texts on statistics, there are statistical tables which included a tabulation of areas enclosed between specific limits as an analyst might want to define them. It should be noted that the distribution is always symmetrical about the mean. In other words, if one is using the 1.96 levels 5% of the results will be outside of the range of u +/- 1.96, but only 2.5% of all results will exceed u + 1.96 and 2.5% of the results will be less than u - 1.96.

Focusing this into a discussion more pertinent to the laboratory and, perhaps more viable with respect to occurrences within the laboratory, let us discuss the rare exception in which an analyst is taking 20 tests on a particular sample using the 1.96 level. Considering that 5% of the results will lie outside that level, the analyst has 1 chance in 20 of missing the true value outside the stated confidence range. At the same time one can decrease this chance by increasing the allowable range. For instance, if the range is R =/- 2.58 the results will be included on 99% of the occasions or 99% of the tests. However, by increasing the confidence limit, one is also increasing the uncertainty in the true value. In this case, uncertainty can be decreased by taking the mean of several analytical results or by decreasing the value.

These statistical concepts allow valuable quantification of the random error of an analytical result and emphasize that decisions, based on the significance of the result, have some risk of being wrong. Knowledge of the standard deviation, of the results is, therefore, vital in reaching objective decisions. Use of the standard deviation will be explained in the following sections dealing with data handling and validation.

9.4.2 Data Handling, Reporting, Recordkeeping

A flow diagram, Figure 1, delineates the original and procedural steps in data generation.

The initiation of an analysis starts with the completion of a project approval form.

The information is computer entered. The computer entry internally creates a report form and inventories the analysis by parameter or compound. The computer entry function of all analytical work requests is a shared responsibility of the sample coordinator and data coordinator. A copy of the analysis request form is manually inserted into a three ring binder notebook for laboratory reference use. The maintenance of the laboratory job reference notebook is a responsibility of the sample coordinator. The group leader/supervisors requests from the data coordinator (D.C.), the computer generated analytical bench sheets for a given parameter each morning or the prior day. The samples and parameters testing sequence is dictated by a weekly work schedule. The weekly work schedule is developed manually each week by the group leaders/area supervisors and approved each week by the laboratory manager. The schedule is developed from a computer printout that inventories and ages by project job or parameter. Contractual due dates and sample holding times are the compliance criteria by which all schedules are judged.

The bench sheets examples are shown in Figure 7, 8, 9. The bench sheets identify to an analyst the proper samples to analyze that day. The analyst lab notebook and the bench sheets constitute the two raw data reporting locations. The content of the laboratory notebook is defined in an earlier section, 7.3.7. The analyst completes the benchsheet information, attaches a drawn calibration curve and follows the analytical sample sequence identified in section 10.0. The analyst identifies which sample(s) were utilized for precision and accuracy determinations. The analyst will assess the data set as being in control or not. The assessment will be described in the data validation section to follow. The analyst will submit to respective group leaders or supervisors all of the abovementioned data and a written statement that the data set is in control for their review. An approved data set is signed off and the group leaders/supervisors transfer the approved data to all appropriate worksheets in the laboratory job reference notebook. The bench sheets and calibration curves are permanently stored. The last entry into the worksheet constitutes a completed project subject to computer generation of a preliminary report. The group leader/supervisors provide the DC with the approved worksheets for computer entry and preliminary report generation. The remaining activities related to preliminary report, final report generation and review and project filing are identified in this manual under sections 7.3.14, 7.3.15 and 7.3.16 respectively.

9.5 DATA VALIDATION

The data validation process includes a set of computerized and manual checks at various appropriate levels of the measurement process.

The data validation process starts with the laboratory analyst. The analyst verify in their

lab notebook that all method specific operational parameters are utilized or met. This information is specifically documented in all instrument logbooks. The analyst then verifies that the calibration of the equipment is linear and documents this in the instrument logbooks. If the operating parameters of a particular method are modified, it should be written in the analyst lab notebook and approved via signature by the group leader/supervisor in the lab notebook. A non-calibrated system must be identified by the analyst and corrections made to achieve calibration prior to sample analysis.

The generation of sample data by an analyst will include the generation of quality control data for each sample set. The monitoring of method blanks, sample spikes, method spikes and sample duplicate analysis is accomplished by the utilization of Schwart Quality Assurance Charts. All quality control data is entered on the precision and accuracy data summary form, Figure 11a. The analyst computes the data precision and accuracy and compares the computed value to the acceptance intervals identifies on the form for that parameter, method, and matrix. The computed value will be determined in control if it lies within the acceptance interval. If the computed value is deemed out-of-control the data set is not submitted for supervisor approval but is brought immediately to the attention of the supervisor and quality assurance officer that an out-of-control condition exists. Jointly, a review is conducted to determine the cause(s) and conduct corrective action. The data set is rerun once the corrective actions have taken place and the new data reviewed as stated above.

The DC receives all the completed precision and accuracy data summary forms and enters the data into the laboratory quality control computer system. The system produces summary reports each day of all quality control data generated for review by the quality assurance officer. The computer system also generates all Schwart Control Charts for method blanks, method spikes, sample duplicates and sample spikes. The charts are permanently maintained and reviewed each week by the group leader/supervisor and the quality assurance officer. The weekly generated charts provide an accurate review of all recently (last 30) qc data points and allows the monitoring of data trends or other anomalies to the system.

10.0 GENERAL QUALITY CONTROL PRACTICES

10.0 GENERAL QUALITY CONTROL PRACTICES

The Quality Assurance/Quality Assurance practices at EARTH TECH are based on several of the following government guidelines:

- "Handbook for Analytical Quality Assurance in Water and Wastewater Laboratories "EPA 600/4-79-019, March 3, 1979
- The Guidelines Establishing Test Procedures for the Analysis of Pollutants Under the Clean Water Act 40 CFR; July, 1990.
- Manual of Analytical Methods for the Analysis of Pesticides in Humans and Environmental Samples" EPA 600/8-80-038 June 1980.
- ASTM
- Test methods for evaluating a solid waste; USEPA SW-846; Third Edition, Revision 0.
- 10.1 The quality control types normally analyzed during sample analysis includes the following: Initial Calibration Blank (ICB), Initial Calibration Verification (ICV), Method Preparation Blank (MPB), Laboratory Control Sample (LCS), Sample Matrix Spike Duplicate (MSD), Continuing Calibration Verification (CCV) and Continuing Calibration Blank (CCB).
- 10.2 The frequency of which these QA types are performed during the analytical run is usually stated within the analytical method. The general frequency over-all of these types, and their respective order within the analytical run is as follows: (following instrument calibration).

Туре	Frequency
Initial Calibration Blank	1-per batch
Initial Calibration Standard	1-per batch
Sample #1	
Sample #2	***
Sample #10	***
Method Preparation Blank	1-per batch
Laboratory Control Sample	1-per batch
Sample Matrix Spike	10%
Sample Matrix Duplicate	10%
Continuing Calibration Blank	10%
Continuing Calibration Verification	10%

Any high level concentrations of analyte will be followed by a blank.

- 10.3 The level of internal laboratory quality assurance effort for the following is divided into 4 different categories:
 - 1. Routine Analytical Services (RAS). No special reporting requirements are required.
 - 2. Reportable Analytical Services (REP). For this type, batch quality control is reported for all analytes.
 - 3. Special Analytical Services (SAS). Each matrix type for a particular submittal will have internal QA performed on these particular samples at the appropriate method frequency.
 - 4. Quality Assurance Project Plan (QAPP). This level of QA encompasses all aspects of the SAS type with full data deliverables similar to CLP reporting packages.
- 10.4 The fundamental QA objective with impacting accuracy, precision and sensitivity of laboratory analytical data is to achieve the QA acceptance criteria established for each analytical method and matrix type.

The control limits established for each method are based on ± 3 standard deviations from the analytical mean. Also encompassed are method advisory limits if provided within the analytical methodologies.

The standard operating procedures that would lead to an outlier being identified and the resulting corrective actions is described in section 9.0, Data Reporting, Validation and Handling. In general, if an out-of-control result occurs the analyst will identify it as such and report the occurrence to the Group Leader and/or Area Supervisor. The Group Leader and/or Area Supervisor will review the data with the analyst to identify the problem, implement a corrective action(s) and then re-analyze the sample(s). The Group Leader and/or Area Supervisor will report the out-of-control occurrence to the Quality Assurance Supervisor that day in writing (Figure 13). The corrective action(s) will be identified in the analyst notebook and in writing to the QA Supervisor.

FIGURE 13

Analytical Quality Control Occurrence Report

Parameter:	· .
Method:	·
Date:	
Analyst:	
Description of Occurrence:	·
Analysis of Occurrence:	
	·
~ ~	
Disposition of Data:	
	·

05-SEP-91		INOF	RGANIC BENCH	SHEET	AUTOMATE	ED CHEMI	STRY
Test#: 389.01-245.01 Parameter: CHLORIDE Method: CL/TRAACS/UTR Ref. Cit.: USEPA-325.2	OE	DL: 2.0 it: mg/l		1. 2. 3. 4. 5.	STD VAL	OBS VAL	NUMBER
Comments:							
ClientSample CO	c gc	Wt/dil factor	Reported Conc.		Spike Oty		EXC DHR
ICB:					I XXXXXXXX I XXXXXXXX		
ICV: Stk Rumpke of Indiana, Inc. 231- 7 1701 YE Rumpke of Indiana, Inc. 231- 7 1702 YE Rumpke of Indiana, Inc. 231- 7 1703 YE Nor-Am Chemical Co. 411- 1 1369 Nor-Am Chemical Co. 411- 1 1370 Nor-Am Chemical Co. 411- 1 1371 Nor-Am Chemical Co. 411- 1 1371 Nor-Am Chemical Co. 411- 1 1371 Nor-Am Chemical Co. 411- 1 1371	B RAB						, i i
MPB:		د اللهو چون متحدد جديد اللهواهية ليسية باست السيد يسويت مي ما اللهوا الله اللهواهية اللهواهية اللهواهية اللهواهية اللهواهية اللهواهية اللهواهية اللهواهية اللهواهية اللهوا			XXXXXXXX XXXXXXXX		
LCS; Stk	}-			.			
SPK: Stk Smp					XXXXXXX		
DUP: Smp	-	، مند <u>سه م</u> ند الله سور عن شد البر سه -		.j	I XXXXXXXX I XXXXXXXX	1	
CCB:	-				İXXXXXXXX		
CCV: Stk	i		i	i	i	į i	

	PAGE 1
Instrument #: Benchsheet ID: Owner:	2463
Date run: Supervisor: Est anal hrs:	.8
Act anal hrs: Samples in batch: Stock std #:	-8
Wavelength (nm): Cell path (mm):	

- injector temperature
- · detector temperature
- ion chamber voltage (GCMS)

7.3.9 Instrument Maintenance Log

The instrument maintenance log is a bound and paginated log which is used to track potential maintenance problems. The log is used every time the instrument is used but may contain several entries on one page. Entries on days where calibrations are correct may be as simple as "calibration met requirements". Anytime the instrument is repaired or modified in any way, the event must be noted with all specifics, including what was done, by whom, and why. A two detector GC has one log tracking, two detectors.

7.3.10 Oven, Refrigerator and Freezer Temperature Logs

Each oven, insulator or furnace, plus all cold storage devices, will have their temperatures checked and recorded daily, or at a minimum, 5 days a week. Each device will have a thermometer in place or a temperature recorder in-place which will be checked by the Data Coordinator. A bound log book with 31 entries will be used to record all entries for each device upon which the DC will record the date and temperature and will initial the entry. The DC will have an NBS thermometer which will move between devices to act as a QA check for the primary temperature device. The log will include the second temperature when measured monthly.

7.3.11 Balance Logs

An Area Analyst will check all balances in the laboratory every day (or at least 5 days a week) using NBS class S weights. The analyst will record each day's reading in a log developed to handle every balance. A balance which fails to meet criteria will be removed from service until repaired. The DC will insure that every balance is serviced and calibrated annually recording such service in the log.

7.3.12 Standard Record Books

Every standard used in the laboratory must be labeled and the label will possess the following information:

- The analyte or analytes contained in the standard
- The concentration
- The solvent
- The preservative, i.e. nitric acid

- The date made
- The Standard Reference Number

The last item, Standard Reference Number, is the identified standard and dilution sequence no. taken from the Standard Record Book in which the standard solution data is recorded.

All standards (including dilutions) will be recorded in a Standard Record Book assigned to the work station. Two record books will be used, each of which has a different purpose. The record books are subtitled as follows:

7.3.12.1 STOCK STANDARDS LOG

This book contains standards starting with the identification of the starting material. One standard and/or standard mix with it's corresponding dilutions are identified.

7.3.12.2 WORKING STANDARDS LOG

A working standard reference number is assigned and the corresponding dilutions are identified.

7.3.13 Control Charts

Each analytical method will require at least one control chart. Some tests may involve several control charts, i.e. duplicate, matrix spikes and method spikes. The QA coordinator will supply the limits to be used to the work station involved. Every data point generated with every analytical batch will be plotted on the chart. Every out-of-control data point will be noted and an action indicated as to the disposition of the data. Completed control charts will be turned in to the DC for permanent change.

7.3.14 Preliminary Reports

After all data has been entered for a project, the computer will flag a project ready for a preliminary report. The report will be identical to the final report in content except for the following:

- Preliminary Report will be reviewed and corrected if necessary on each page in large type.
- Comments necessary to the project will be printed under each sample or at the end of the report.

The DC will print the preliminary report and issue a copy along with the project file to the lab supervisor for review and corrections. The supervisor will sign off on the preliminary report after including comments, if appropriate, indicating that corrections are necessary. Afterwards, the supervisor(s) will pass the preliminary report to the QC Supervisor (QC) who will review and correct the report including a signature and comment. The QC will return the preliminary report and file to the DC. The DC will make all corrections as required and review report structure for completeness. If no corrections are required, the DC will sign and date the preliminary report and place it in the Project File. The DC will then print a Final Report. When corrections are necessary, the DC will execute all corrections and indicate such changes on the initial preliminary, which is then filed in the project file. A new preliminary is then printed and issued for review.

7.3.15 Final Report

After the preliminary report has been corrected and cleared all reviews, the DC will manually alter the computer flag and print a Final Report which will be placed in the project file folder and forwarded to the AM for approval. Space will be provided on the c.o.c. project file folder for the signatures of the Analytical Manager, the QA Supervisor and the Project Manager, all of whom are thus certifying that the report is complete, correct and defensible. The DC will then arrange for delivery of the final report.

7.3.16 Project Files

The Project File is the comprehensive record of every project completed at EARTH TECH. A project file initially consists of a file folder set up by the Lab Secretary (LC) at the time of log-in. Chain-of-Custody projects will be stored in a locked COC file with strict limited access while routine project files are stored in a separate nominally limited access file. The LS will be responsible for including the following in the project file:

- Project Sheets
- Project Approval Sheets (if applicable)
- Problem Project Sheets
- Chain-of-Custody Forms
- All correspondence or documents received with the samples
- Preliminary Reports
- Separate Report Papers, i.e. Field Reports (if applicable) Final Report
- Any additional paperwork which may follow the report

All project files are stored for a period of 4 years.

8.0 SAMPLE CONTROL, FLOW, AND STORAGE

8.0 SAMPLE CONTROL, FLOW, AND STORAGE

All samples received by EARTH TECH Engineering and Sciences must be logged in before any work is performed on the samples. The purpose of the log-in procedure, including sequential numbers assigned to all samples received in the facility, is to insure that EARTH TECH has a means by which samples can be tracked, data can be stored, and quality control can be tracked for any sequence of events during a particular analytical period. In handling projects in this manner, EARTH TECH, or the client, can insure a consistent and documented sequence of events under any analytical situation.

Management acknowledges that there are situations in which log-in of samples will be difficult due to rapid turn around requirements for particular compounds that may decompose or volatilize. An example of this kind of analysis is the total coliform samples which can be anticipated and for which holding times are short. The project approval form discussed within this manual will make it possible to preassign project numbers to samples arriving at the facility. Should a secondary mode of operation be necessary for the receipt of such samples, a mechanism will be developed between the sample coordinator and the Quality Assurance Supervisor. Any deviation from the standard log-in procedures detailed herein will be at the discretion of the laboratory supervisor or the laboratory manager. The execution of the log-in procedures for Chain-of-custody samples (see Section 8.8) is extremely crucial. Samples, that have been designated for Chain-of-Custody by a client, possess the potential of involvement in litigation or other legal situations., i.e. standards development or patents. By breaking Chain-of-Custody requirements, all results are invalid for such purposes.

8.1 PROJECT INFORMATION

All information relative to a specific project must be recorded on a project approved form by the manager responsible for that project prior to the receipt and log-in of samples. Projects, and therefore samples which are not routine to the EARTH TECH laboratory, must have prior approval via the New Project Approval Form before samples may be received.

8.2 NEW PROJECT APPROVAL

The project approval form include the following information:

- Client name, address, and client contact personnel
- Compound names or computer test codes or group computer test code
- Project and sample comments
- · Contract number or purchase order for project
- Instructions relative to the proper completion of the project
- Pricing information relative to the proper completion of the project
- Chain-of-custody requirements

- Specific report requirements
- · Additional requirements such as rush, hazardous, labile

8.3 NEW PROJECT APPROVAL

If a new project will require support from the analytical facilities, that project must be approved by the laboratory supervisors and the laboratory manager prior to project pricing and sample receipt. Routine samples are those samples and analyses which are continuously processed by EARTH TECH, such as priority pollutant samples, microbiological samples, and drinking water samples.

Projects which are non-routine are those that may require special testing, or which request parameters not routinely run within the laboratory, special holding times, or rush turn around. Non-routine projects will require that a New Project Approval Form be completed which includes the signatures of all the parties involved with the project. For example, if specific physical testing is necessary, the supervisor of the physical testing facility and the laboratory manager will have to sign off on the form thereby agreeing, not only to the project content, but for the turn around, the report requirements, the detection limits and the quality control reports that may be necessary to properly carry out the project requirements. Projects and/or samples arriving at EARTH TECH which are nonroutine in nature, and for which there is no signed Project Approval For,, will not be processed. In this case, the manager responsible for the non-routine project will be advised of the problem and will then explain to the client why the delay is necessary for the execution of testing before proceeding to obtain the necessary approvals. The Project Approval Form must be completed and signed by all parties prior to the start of log-in.

8.4 SAMPLE RECEIPT

8.4.1 Introduction

All samples will be received at the EARTH TECH Laboratory by the Sample Coordinator (SC). The job description for the Sample Coordinator is attached as Figure 10. It will be the responsibility of the SC to determine: a) whether or not the proper project sheet is available for the arriving samples; b) whether or not the samples require chain of custody; c) whether or not the samples are labile in nature and require immediate attention; d) the manner in which those samples will be split, preserved and stored or routed. It is the objective of the SC to insure that the receipt of all samples is consistent with the requirements of the EARTH TECH Manual and that all pertinent information relative to those samples is recorded. This information may be used in client reports, communicated to the laboratory or to the client and, in some cases, reported to a legal authority relative to Chain-of-Custody samples.

FIGURE 10

SPECIFIC RESPONSIBILITIES

The SC's duties and responsibilities shall include, but not be limited to:

- 1. Sample receipt.
- 2. Insuring that COC sample receipt includes shipper's signature on COC forms.
- 3. Inspection of sample shipping containers for presence/absence and condition of:
 - a) custody seals, locks, "evidence tape", etc.
 - b) container breakage and/or container integrity
- 4. Recording conditions of both shipping containers and sample containers (bottles, jars, cans, etc.) in appropriate logbooks or on appropriate forms.
- 5. Signing appropriate documents shipped with samples (i.e., Chain-of-custody record(s).
- 6. Verifying and recording agreement or non-agreement of information on sample documents (i.e., separate tags, Chain-of-Custody records, traffic reports, airbills, etc.) on appropriate forms and on the EARTH TECH project sheet.
- 7. Initiating the sample and project log-in procedures on appropriate laboratory documents and according to the standard operating procedure, including the initiation of project files with sample control records.
- 8. Marking or labeling samples with laboratory sample numbers, as appropriate.
- 9. Placing samples and spent samples into appropriate storage and/or secure areas.
- 10. Controlling access to samples in storage and assuring that laboratory operating procedures are followed when samples are removed from and returned to storage.
- 11. Monitoring storage conditions for proper sample preservation such as refrigeration temperature and prevention of cross-contamination.
- 12. Returning shipping containers to the proper client or licensed disposal facility.
- 13. Providing for the splitting of samples into required aliquots, including preservation for each working station.

8.4.2 Examination of Shipping Container

Immediately upon receipt of a sample shipment at the EARTH TECH Laboratory, the SC will examine the shipping container (the container may be a box, a cooler, a styrofoam container, etc.) to ascertain and document the condition of the samples and to process Chain-of-Custody papers, where appropriate. The SC will record the condition of the shipping container, the identification of the shipper, the presence or absence of any seals on the container (if it is Chain-of-Custody), and the labeling which may include special instructions prior to opening the container. If the shipping container is damaged, a report will be sent immediately to the shipper and the lab supervisor (see Section 8.15.2, Problem Project Sheet).

8.4.3 Carrier Sign Off for Chain-of-Custody Container

Should the SC identify the shipping container as being a Chain-of-Custody container, the SC will attempt to have the carrier's representative sign off on the Chain-of-Custody papers which should be available either on the outside of the shipping container, or immediately inside. An example of a Chain-of-Custody record is attached as Figure 5, (Section 7). In the event that the carrier's representative is unwilling to cooperate in this fashion, the SC will identify, in the proper position on the Chain-of-Custody document, the shipment number, the date of receipt, and sign off, attaching a copy of the shipping log for that particular container.

8.5 EXAMINATION OF CONTAINER CONTENTS

Unless the shipping container contents are marked "hazardous" the SC will proceed to open the sample container. If the SC had not previously identified the project sheet appropriate for these samples, the SC will attempt to ascertain immediately the origin of the samples found in this container and obtain the appropriate project sheet. If a project sheet is not found, the SC will lock up the samples and notify the lab manager as described in Section 2.0. The SC will identify whether or not all the samples have arrived intact, whether or not the labels are intact and attached properly, and whether or not the samples have leaked in any fashion. The SC will also identify any shipping instructions, field instructions, or any other materials that may be present in the shipping container.

8.5.1 Chain-of-Custody Shipments

Should the SC identify the shipping container as a Chain-of-Custody project, the SC will immediately follow the procedure outlined in Section 4.0, "Chain-of-Custody Samples".

8.6 PROJECT VERIFICATION

The sample coordinator, having opened the shipping container and examined all the samples, will verify that the project sheet matches the samples, the number of samples received is consistent with the project sheet, and that the requirements identified on the project sheet are consistent with any paperwork obtained which will include the project sheet and any other documents in the sample container. The project files will be kept by the SC in a locked filing cabinet. If all required project information is not complete, the SC will fill out a Problem Project Sheet (see Section 5.2) and turn it over to the Project Manager.

8.7 LABILE SAMPLE DISTRIBUTION

Should the SC identify labile samples within the shipping container, (i.e. coliforms or nitrites) for which there is a very short holding time and a need to rapidly move the samples into the laboratory, the SC will make every effort to immediately log-in those Should log-in be delayed, the SC will coordinate with the responsible samples. analytical group in order to move the samples into analysis. The coordinated effort will included means by which the SC can label the samples after log-in and insure that the results correlate with the proper samples. The SC will provide computer generated sample identification to the responsible analytical group. It will be the responsibility of the SC, once labile samples have been distributed to the laboratory to insure that those samples are properly logged in and that they are labeled with properly sequenced numbers. The agreement that is made between the SC and the appropriate laboratory manage or laboratory supervisor will be based on the premise that the SC understands that he/she is ultimately responsible and will be held accountable for any samples that are lost in such a movement. Consequently, the SC will find the samples that are labile and apply the necessary labels.

If a shipping container is labeled "Hazardous", the SC will immediately notify the laboratory supervisor who will determine the extent of hazard and/or the manner in which the samples will be handled. The supervisor will involve the laboratory manager as needed in resolving questions of hazardous samples.

FIGURE 11

POSITION DESCRIPTION FOR SAMPLE COORDINATOR

GENERAL

The Sample Coordinator (SC) is responsible for the receipt, log-in, and storage of all client samples at EARTH TECH. The SC is responsible for the receipt, storage and custody of all Chain-of-Custody (COC) samples including distribution of COC samples to lab personnel per EARTH TECH COC procedures (section 4.0, EARTH TECH Log-in Procedure). In order to ensure the successful analyses of samples, it is critical that the SC obtain and communicate to Project Manager, lab supervisors, and lab personnel, all information necessary for the processing interpretation and reporting of all samples analyzed.

QUALIFICATIONS

High School Diploma and a minimum of 2 years of college or equivalent. A knowledge of chemistry and testing procedures helpful. Excellent verbal, written and organization skills, including a propensity for detail necessary for successful completion of job.

REPORTING RELATIONSHIPS

The SC will report to the laboratory manager. The SC will communicate closely with the Director and Project Managers to obtain project information.

8.8 CHAIN-OF-CUSTODY SAMPLES

8.8.1 Continuance of Log-In Procedures for Chain-of-Custody Samples

All samples in the possession of the EARTH TECH Laboratory under Chain-of-Custody (COC), must be traceable from the time the samples are received at the EARTH TECH door (or collected by EARTH TECH staff) until results are reported and sample disposition has been determined from the client. For any samples that may be collected during enforcement investigations, under litigatory requirements, or evidentiary in nature, Chain-of-Custody procedures are required.

8.8.2 Examination of Container Contents

Although Section 8.4.2 under Sample Receipt discusses the thorough examination of container contents, the proper examination of a container which is involved in a Chain-of-Custody procedure is even more important. For example, should the sample labels be mismarked or a particular sample to somewhat strange in nature, it is necessary to note all observations and deviations from the project sheet. It is better to be overly observant than to allow possible anomalies to go unnoticed. It is the SC's responsibility to examine whether or not each of the sample containers are individually sealed, whether those seals are intact, whether a sampler's initials are on the seals, and whether or not the paperwork matches the contents of the package. In addition, the SC must note whether or not all the dates and times are consistent, and whether or not the sample description on the paper work matches the description on the sample container.

8.9 PROJECT VERIFICATION

In the same manner in which the examination of the container contents is critical to a COC project, the verification of the project is equally important. These project verification steps include not only the need to follow the requirements identified in Section 8.6, but also thorough examination of all aspects of the project and the consistency of all the paper work involved with those particular samples in that shipping container. It is also important that the SC place in the COC project file: the shipping document; a signed Chain-of-Custody document including the sign off from the shipper's representative (See Section 8.4.3); a copy of the project sheet; a copy of the Project Approval Form is appropriate; a copy of the filed sampling report if appropriate; and originals of all paperwork received for the project. The COC project file is kept in locked storage in the possession of the SC.

8.10 CHAIN-OF-CUSTODY LOG-IN

The log-in procedure identified in section 8.15 titled "Log-in", is followed in the same manner for Chain-of-Custody samples with a few modifications. Those areas which are changed are addressed in the following sections:

- Sample Storage
- · Project Files
- Laboratory Access
- Data Storage

8.11 CHAIN-OF-CUSTODY SAMPLE STORAGE

All samples received under Chain-of-Custody procedures will be kept under locked storage and will be distributed for analysis to the laboratory only when the analyst has signed for the samples on the form shown in Figure 6, (Section 7). The SC or a designated representative will provide access to COC storage. Records of movement of all COC samples within the lab facility must be recorded.

8.12 CHAIN-OF-CUSTODY PROJECT FILES

All Chain-of-Custody project files will be kept in a project folder in a locked cabinet with all related documents and paperwork relative to those files.

8.13 MAINTENANCE OF LAB CUSTODY

Laboratory custody must be consistent with all the Chain-of-Custody requirements from the beginning of sampling to the final report. To this end, every analyst requiring access to the Chain-of-Custody samples will go to the SC for access to the COC locked sample storage. The SC will insure that the analyst signs for the receipt of all COC samples on the form shown in Figure 6, (Section 7) and that the analyst returns and signs in those same samples on the same day for which they were signed out. This documentation, after the completion of all analyses, will be placed in the locked Chain-of-Custody project file by the SC.

8.13.1 Sample Custodian

The COC sample custodian at the EARTH TECH Laboratory will be designated as the Sample Coordinator (SC). The SC is responsible for following the COC requirements outlined in these procedures for all samples received.

8.13.2 Lab Custodial Responsibilities

It will be the responsibility of every analyst signing for a Chain-of-Custody sample or samples to insure that; a) these samples are kept in a minimum access

facility; b) they are within their possession during the particular period during which they are being analyzed; and c) the analyst returns those samples to the Chain-of-Custody lockup in the manner prescribed. The analyst will sign out and return the samples to COC lock-up on the same day. The analyst will be using the SC as the sample custodian for all COC samples. Due to the legal implications for the client of breaking the COC procedures and possibility of legal action that could be taken against EARTH TECH, errors in the execution of Chain-of-Custody procedures will not be tolerated.

8.14 CHAIN-OF-CUSTODY SAMPLE DISPOSAL

All samples received for COC procedures will be stored in the EARTH TECH Laboratory COC lock-up facilities until a final report is issued. It will be the responsibility of the Project Manager, in cooperation with the SC, to obtain information from the client relative to the length of time the COC samples will be stored. It is anticipated that for long term storage, i.e. more than 30 days, the client will reimburse the EARTH TECH Laboratory an appropriate rate for keeping completed samples under Chain-of-Custody procedures. No Chain-of-Custody samples may be discarded until written permission is received from the client relative to disposal of those samples.

8.15 LOG-IN

8.15.1 Introduction

After the SC has inspected the shipping containers, the project sheets, the samples and any documentation required in Sections 8.4 and 8.8, the SC will insure that all pertinent information is entered on the project sheet. There are specific areas of the project sheet that are to be completed by the SC, i.e., date and time received. The EARTH TECH project sheet is included as Figure 2, (Section 7).

Minimum information required for log-in include:

- Client's name and Client contact, as well as client #, is assigned.
- The due date
- The analytical test or test codes or group tests
- Specific project comments
- Contract requirements
- Contract number
- Pricing if necessary
- The approval for non-routine projects
- Chains-of-Custody, if required
- Specific report requirements

8.15.2 Project Problems

If any of the information identified in sub-section 8.15.1 is missing, the SC will immediately notify the Project Manager, via a Problem Project Sheet, Figure 4, (Section 7) of the discrepancy. The Project Manager will make all reasonable efforts to insure that the answers are provided to the SC immediately.

Simple Project Sheet deficiencies such as client number, extra comments, or the contract number, should not prevent log-in. The SC will proceed with log-in addressing the unknowns as subjects that must be changed or modified once the information is received. It is the responsibility of the SC to log-in all samples as received at EARTH TECH whenever possible.

8.15.3 Samples on Hold

When there is a considerable amount of inadequate information on a project sheet, i.e. a missing test, or broken samples, the entire project will be placed on hold until the information is available or the corrective actions have been taken to insure that NSF is not held responsible for a poorly handled project. The SC will notify the Project Manager via a Problem Project Sheet as to the hold status of the project and the reasons for the hold. The Project Manager will make every attempt to quickly identify the necessary actions that will be taken for those samples or the remaining samples for that project. The Project Manager may approve log-in of the remaining samples for a portion of the project in order to insure that the project progresses. Projects that are placed on hold will be locked in a "project hold" area, (like the Chain-of-Custody sample storage area) so that those samples are not lost or confused within the system. The SC will insure that those samples are retrieved and logged in as soon as the appropriate changes have been made and the samples are freed for log-in.

8.15.4 Handling Labile Samples

All samples received by the SC that are labile in nature, i.e. coliforms, need to be logged into the facility in a very rapid fashion in order that they may attended to within the analytical holding time. The most labile of all samples are the microbiological samples, which must be forwarded to the micro lab as soon as possible. The SC and the Project Managers responsible for micro work will attempt to insure that appropriate information is available to the SC in order that the SC can assign numbers for all labile samples. These numbers can be assigned in advance and samples may be logged into the system as soon as they are received. Samples such as nitrites, which are labile but have a somewhat longer holding time, will usually be logged into the system like normal samples. However, slow shipment or other problems may require the lab to initiate the analyses immediately. In such a case, assuming a project sheet was initiated in

advance o sample receipt, the SC can assign laboratory in an expedient fashion. The SC will make all efforts to insure that samples move through the laboratory in a timely fashion when holding times are of utmost importance to the proper completion of the analytical requirements.

8.16 COMPUTER LOG-IN

It is anticipated that all samples received at the EARTH TECH Laboratory will be logged on to the computer by the SC. The computer assigns a sequential number to every sample. Additional codes such as the month and the year of the samples may be added in front of the sequential number for continuous identification of these samples. The SC will have the computer generate these sequential numbers for each sample in every project. A project identifier will be printed on the labels which are attached to every sample and every aliquot of a sample.

8.17 SAMPLE SPLITTING FOR THE CHEMICAL LABORATORY

The EARTH TECH Laboratory Chemist Project will inspect to insure that all samples received at the EARTH TECH facility are received in the appropriate containers with the correct preservatives (Samples which must be split at log-in are subject to added error). The labels and the appropriate preservatives are depicted in Figure 12.

8.17.1 Bottles and Preservative Requirements

The EARTH TECH Laboratory has a series of bottle and preservative requirements that must be met before the log-in of samples into the laboratory. In the event that EARTH TECH is unable to provide sample bottles, or circumstances prevent the splitting of samples in the field, the SC will provide sample splitting services. These services will include taking the sample as received and subsampling it into the appropriate bottle and preservative requirements as set forward on the attached list of bottle and preservative requirements.

8.17.2 Inorganic Samples

The SC will insure that sufficient sample volume is available before initiating the splitting of a sample. If uncertain, the SC will involve the laboratory supervisors in order to insure that all areas of the lab have sufficient samples. In the event that sufficient samples does not exist, the SC will identify the sample as a problem and will notify the Project Manager immediately for resolution. The sample will be logged in only after a resolution has been reached.

8.17.3 Organic Analysis

When a bulk sample arrives for organic/inorganic analysis and sufficient sample exists, the SC will transfer the sample to the organic preparation supervisor who will split the organic aliquots and return all aliquots to the SC. The remaining sample will then be returned to the SC who will split off the inorganic aliquots into the proper preserved containers.

8.17.4 Solid Samples Splitting

When solid samples, such as sediment or soil, are to be received at EARTH TECH, every attempt will be made by the Project Manager and field sampling personnel to insure that two samples are provided as replicates for the appropriate tests. One of these samples will be assigned to the organic facility; the other will be assigned to the inorganics facility. If only one sample is received and if organic analyses are required, the organics preparation chemist will be responsible for the initial splitting of the sample. Solid samples will be made homogeneous by either one or all of the following manners:

- Stirring especially when volatile organic analytes are required
- Air Drying and Grinding
- Particle separation (Sieving)
- · Quartering by ASTM Procedures

The lead organic chemist and the SC are responsible for the decisions on how a solid sample will be split. Problems or concerns which may arise on a solid sample will be addressed to the Project Manager and the laboratory manager for resolution. After the organic portions have been removed or split, the remaining sample will be provided to the inorganic facilities for any further splitting they deem necessary.

8.18 SAMPLE LABELING

All samples received at the EARTH TECH Laboratory are labeled by the SC at the time of log-in. These labels will include information such as the requested sample number, the client number if supplied, the contract, the EARTH TECH Laboratory project number, and/or the client. It is anticipated that sequential sample labels will be provided by the computer after the SC has logged the project into the computer.

8.19 DISTRIBUTION AND STORAGE

Logged samples will be taken by the SC to the appropriate walk-in cooler for cold storage or to the room temperature storage area indicated for metals.

COC samples are stored as set forth in Section 4.0.

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EARTH TECH-ANALYT AL SERVICES-GR CHAIN OF DDY

CLIEHT:

Rumpke of Ohio, Inc.

PROJECT: Quarterly Monitoring Cincinnati, Ohio Landfill

SUBMITTAL: August, 1991 Groundwaters PARAMETER: CARBON, TOTAL ORGANIC

HETHOD: TOCZOYTOZUTE

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1285					

Formerly WW Analytical Services

Date Requested:	1	1	Date Due:	1	1	
Dispatched By:						
Project:						
Project Manager:_						
Project No:						
Location:						

Sample Inventory and Master Bottle Packing List

Sample Sample Sub-Portions-Preservative and Tagging Codes Locations Number 1 2 3 4 5 6 7 8 9 10 11 12 13 14 15 16 17 18																										
Locations	Number	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18							
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Indicate Sample Sub Portion with an X

Multiple Sub Portions for the same Bottle Type can be Identified by Entering the Number Needed

NO.	DESCRIPTION	PRESERVATIVE	TAG COLOR	FILTERE
	Waters			
1	40 ml Vial for Purgeable Organics	1+1 HCL Yes / No Cool to 4* C	Yeilaw	
2	1000 ml Amber Glass Non Purgeable Organics	Cool to 4° C	Salmon	
3	mi Plastic - Non Preserved	Cool to 4° C	Green	
4	ml Plastic - Nutrients	pH < 2.0 w/H ₂ SO ₄	Blue	
5	mi Amber Plastic - Cyanides	pH to > 12 w/NaOH	· Light Blue	
6	mi Plastic - Metals	pH to <2 w/HNO ₃	Red	
7	1000 ml Glass - Oil & Grease / TPH	pH to <2 w/H ₂ SO ₄	Dark Blue	
8	125 ml Whirl Pac Bag / Bottle Bacteria	Cool to 4° C	Brown	
9	500 ml Glass - Suifide	0.5 ml Zing Acetase + 0.5 ml NaOH to pH >0	Light Green	
10	250 ml Amber Glass - TOX	pH to < 2 w/H 25O 4 Cool to 4° C	Lilac	
11	40 ml Amber Glass - TOC	pH to < 2 w/H ,SO 4 Cool to 4° C	Pink	
12	2000 ml Plastic - Radiological	pH to < 2 w/HNO ₃	Gray	
13	500 ml Amber Glass - Phenois	pH to < 2 w/H2 SQ	Brown	
14	250 ml Amber Glass - Formaldehyde	Cool to 4° C	Orange	
	Soils			
15	mi Wide Mouth Plastic	Cool to 4° C	White	
16	mi Wide Mouth Amber Glass	Cool to 4° C	Manilla	
17	125 ml Vial for Purgeable Organics in Soil	Cool to 4° C	Light Yellow	
18	Other			

8.20 PROJECT FILES

8.20.1 Routine Project Files

The SC will obtain a manila folder and label that manila folder with the name and number of the project. The folder will indicate the EARTH TECH Laboratory project number or contract number, and Chain-of-custody if applicable. With the agreement of the laboratory supervisor (lead), the project manager, and the laboratory manager, a particular project folder may include a series of projects logged in under sequential numbers. An example would be a daily log-in for the same project for a week or month before a new project folder is generated. It is, however, the responsibility of the SC to insure that all logged projects are filled in a project file folder.

8.20.2 Chain-of-Custody File Folder

The SC, upon logging in any Chain-of-Custody project, will provide the same type of manila folder project file, as discussed in Section 5.7.1, for each project. However, the project folder will be maintained in the locked Chain-of-Custody file and cabinet and will be kept by the sample coordinator.

8.21 SAMPLE STORAGE

8.21.1 Non Chain-of-Custody Storage

The SC, after completing all the log-in processes of various samples connected with a particular project, will store the samples in the designated areas in the EARTH TECH laboratory.

- Routine Water and Solid Samples: Samples which need to be refrigerated will be stored in the walk in facility designated for all routine water and soil samples.
- Routine Volatile Water and Solid Samples: All these samples will be
 placed in the designated VOA refrigerator(s) located within the analytical
 facility. Volatile water and soil samples are segregated and stored
 separately. No other samples or standards may be stored in the VOA
 refrigerator(s).
- Routine Water and Solid Samples for Metal Parameters: The preserved water samples and solid samples, which are not preserved, may be stored on shelves designated for the metals analysis.
- Odoriferous and Hazardous Samples: These samples will be stored in a special vented facility within the laboratory which is designated for Odoriferous and hazardous samples. These samples will be identified to the

lab personnel and noted on the log-in procedures in order to insure that the lab personnel are aware of the problems with these samples.

8.22 CHAIN-OF-CUSTODY SAMPLE STORAGE

All samples that are involved as physical evidence in a legal procedure or simply identified as Chain-of-Custody will be handled under certain procedural safeguards. These safeguards have been tentatively identified in section 4.0 but for purposes or reiteration are again addressed below:

NOTE: For any legal proceedings, the court must be shown that the laboratory is a secured area, that all samples have been stored in a secured fashion, and samples can be accounted for at all times.

8.22.1 Chain-of-Custody Water and Solid Samples

All samples of this nature will be stored within the locked confines of the Analytical Laboratory. Access is only available to authorized personnel.

8.22.2 Water and Soil Samples for Metals

8.23 GENERAL LAB SECURITY

Access to the EARTH TECH Laboratory is handled in a secured fashion restricting entrance only to those people designated as having access to the laboratory facilities. Restricted access applies to all areas in which samples are stored or analysis takes place. It will be the responsibility of all the analysts, as well as the supervisors and the SC, to insure that the safeguards employed, including locked doors and limited access, are followed and maintained at all times.

9.0 DATA HANDLING, REPORTING, RECORDKEEPING AND VALIDATION

9.0 DATA HANDLING, REPORTING, RECORDKEEPING AND VALIDATION

There are two significant aspects of any analytical procedure:

- The selection and use of a method appropriate for the analyte and matrix
- The collection, control and interpretation of the data generated.

Encompassing these two components is the Quality Assurance program. The QA program provides means by which method selection can be validated, the method can be controlled and the appropriate data generated, displayed and reduced.

The following sections deal with error, data handling, data validation, data reporting and data recordkeeping.

9.1 ERROR: IT'S NATURE AND SIMPLE STATISTICAL CONCEPTS

9.1.1 Random Errors

Repeated analysis of identical aliquots of a homogeneous sample does not give a series of equivalent results. The results will differ among themselves and they will be more or less scattered about some value. The scatter can be attributed to random error, so named because the prediction of the sign or magnitude of the error of any particular result is not possible at the time of analysis.

One therefore, says that each result must have an uncertainty attached to it, and can be regarded only as an estimate of the true value. Generally that estimate will differ from the true value. Random errors are caused by uncontrolled and/or uncontrollable random variations in factors which affect analytical results, i.e. variations in the volumes of the reagents added, variations in the concentrations of reagents, variations in the time allotted for the chemical analysis, a contaminated glassware, poor quality reagents, instrumental fluctuations. Among the various texts that are available discussing errors, the terms repeatability, reproducibility and precision have been used to denote the scatter of results. The term "precision" will be used throughout this manual and is the most common term used for random error in this country and especially by the EPA.

Precision does improve as the scatter among results becomes smaller. All analytical results have random error present which necessitates statistical techniques to evaluate the results and to provide correct inferences of the true value of the result.

9.2 SYSTEMATIC ERRORS

Systematic errors are indicated by the tendency of results to be greater or smaller than,

the true value. It is necessary to take care in exactly defining systematic error because the analysis is also subject to random error. The mean of n analytical results on the same sample approaches a definite value u as the number of results increases indefinitely. When u differs from the true value Tau results are said to be subject to systematic error of the magnitude B, wherein B is equal u minus Tau. Bias is the term used synonymously with systematic error and will be used in that fashion throughout this manual. Analytical methods, which are subject to interferences from substances present in the sample, or methods that only recover a fraction of the material present are an example of systematic error.

It is impractical to make an indefinitely large number of analysis on a single sample in order to determine the true value of u is known. At the same time a practically obtained value for a sample that is based on minimal analysis is subject to random error, so that the experimental estimates of bias will also be subject to random error. Therefore, statistical techniques are also required when bias is to be estimated.

The basic difference between random and systematic error is that, in principal, the latter may be predicted so that a correction can be made to eliminate its effect. An example of this allowance can be accounted for in the effect of fluoride in the determination of aluminum by absorbance measurements. This effect is overcome by adding to the calibration standards an amount of fluoride equal to the fluoride content of the sample: The added fluoride in the calibration standards then eliminates the systematic error of fluoride interference. However, it must be recognized that the complete elimination of systematic error may require such detailed knowledge of the properties of the sample that the correction of the analytical system is impractical and would in fact increase the amount of random error. Thus, in all applications where unbiased results are necessary, the approach to be used is to devise and use analytical systems capable of giving results which have negligible systematic error.

9.3 TOTAL ERROR

Some analysts use the term accuracy to denote only systematic error. The term accuracy as applied in this manual will denote total error of the results. In other words, accuracy represents the combined systematic and random error of the results and, therefore, the accuracy of an analysis improves as the total error becomes smaller. For the purposes of visually seeing random and systematic error, Figure 6-1 should be referred to for any easy identification of the various types of error.

9.4 STATISTICAL TECHNIQUES

Statistical techniques are essential to the measurement of analytical error. This manual and this section recognize that many analysts have had little experience with statistical technique. This section is, therefore, written in such a way as to explain simple but basic concepts of the statistical approach and to describe the particular techniques most

Detroit Coke Corporation Detroit, Michigan RCRA Facility Investigation

Quality Assurance Project Plan

Appendix A: TriMatrix Laboratories SOPs

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April 1996

DCC 0104



EPA Method	Detroit Coke QAPP	Delco QAPP
8260/624	GR-04-104 12/26/95	GR-04-104 4/18/96
3510B Modified	GR-09-101 1/18/96	GR-09-101 1/18/96
3550 Modified	GR-09-103 1/18/96	GR-09-103 1/18/96
160.3	GR-07-104 1/10/96	GR-07-104 1/10/96
Organic Extraction Procedures for making Spiking Standards	GR-09-100 2/7/96	None
8270 & 625 Modified	GR-04-102 12/26/95	GR-04-102 4/19/96
7060A & 7740	GR-01-125 4/2/96	GR-01-125 4/2/96
3010A	GR-01-121 1/17/96	GR-01-121 1/17/96
3020A	GR-01-120 1/17/96	GR-01-120 1/17/96
3050A Modified	GR-01-103 2/1/96	GR-01-103 2/1/96
200.7/6010A	GR-01-100 4/4/96	GR-01-100 4/4/96
200.7/6010A	GR-01-101 3/7/96	GR-01-101 3/7/96
200.9 & 7000	GR-01-108 4/3/96	GR-01-108 4/3/96
7470/245.1 Modified	GR-01-123 4/4/96	GR-01-123 4/4/96
7 4 71A	GR-01-109 1/30/96	GR-01-109 1/30/96
#SDM 4500-CN B, 4500 CN C, U.S. EPA 9012 Modified	GR-18-106 1/17/96	GR-18-106 1/17/96
4500-CN-E	GR-05-104 1/17/96	None
335.3/9012 Modified	GR-02-100 1/10/96	GR-02-100 1/10/96
Lachat Quick Chem AE Operation	GR-02-103 1/17/96	GR-02-103 1/17/96

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